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OF THE
AMERICAN LEATHER CHEMISTS ASSOCIATION

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IMPORT - EXPORT

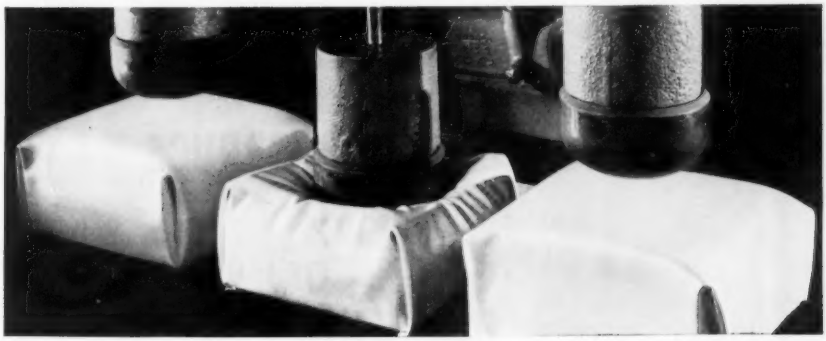
New tanning-mates

give plumper, tighter, brighter

WHITE LEATHER

ZIRCOTAN S / LEUKANOL C

TWO NEW LEATHER CHEMICALS RECENTLY DEVELOPED BY ROHM & HAAS



These white leather samples are being subjected to hundreds of thousands of flexings from pounding weights in a grueling flex test at the Rohm & Haas Leather Research Laboratories.

Now you can get a plumper, tighter, brighter white leather by using ZIRCOTAN S and LEUKANOL C in your tanning formulation. This new tanning combination fills the hide thoroughly, and gives the finished product that *luxury feel* so characteristic of fine upholstery leather.

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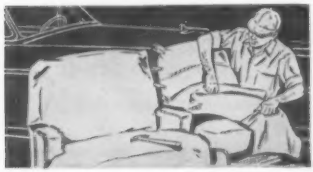
You'll want to know more about our new tanning-mates, ZIRCOTAN S and LEUKANOL C . . . and our many other leather chemicals and services.



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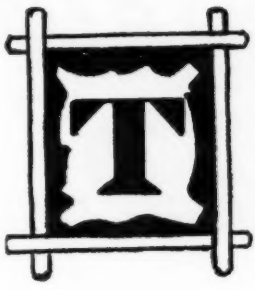
OF ALL DESCRIPTIONS



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"BESTSOLUBL"
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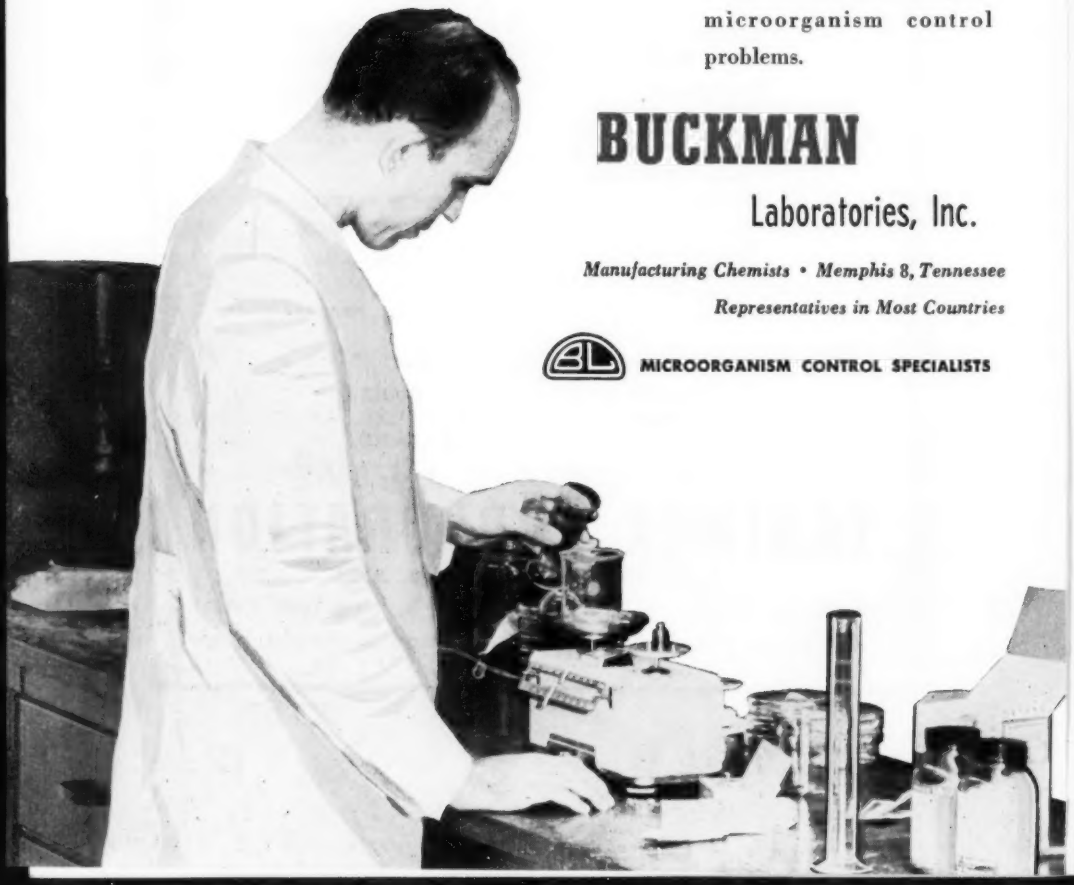

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2 to 1

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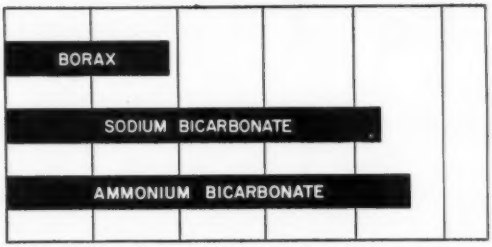
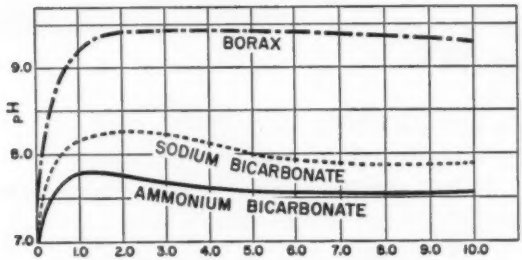


Pounds of hydrochloric acid neutralized by 1 lb. of alka.i

at Lower pH for



Comparative pH of solutions at various concentrations

Superior Leather with SOLVAY Ammonium Bicarbonate

Here's how SOLVAY Ammonium Bicarbonate's unique neutralizing action can lead to superior leather. By penetrating deeply and uniformly it provides the superior neutralizing action which assures more uniform dyeing, more beautiful grain and finish. Try SOLVAY Ammonium Bicarbonate in the economical new 100 lb. vapor-barrier paper bags.

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- used as a leather pretan for better break and smoother grain
- bleaching chrome tannages for white leathers
- syntan-vegetable tan staining, or retanning of garment suede leathers to be crusted
- dye leveling and mordanting
- shearling pretannage

ARKOTAN® B

- used like Arkotan but for fuller leathers with tighter break, smooth grain and even color
- permits greater penetration of the tannning extracts, better take-up in bark retanned chrome leathers and full bark tanned leathers; reduces sludge in bath when solid quebracho is used
- contains a metal sequestering agent to aid tanning in water with high mineral content

ARKOTAN® N

- neutral syntan for pretanning of soft, mellow leathers for fine grain and smooth, well let out appearance and feel
- produces level color with minimum loss of color value in dye leveling and mordanting

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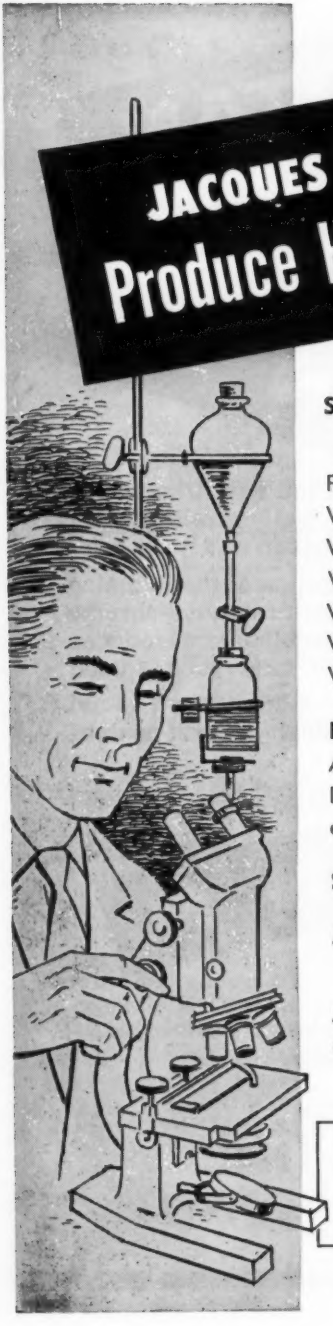

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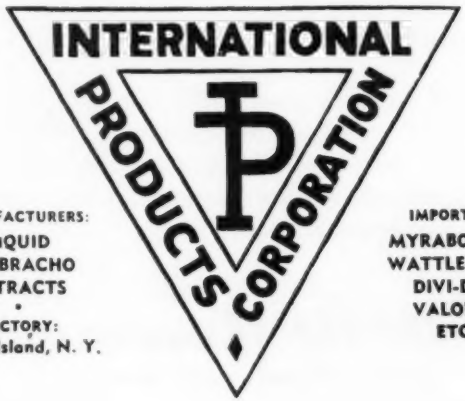

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Sodium Sulfide
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You can get this built-in alkalinity control the next time you order, by specifying Hooker flake sodium sulfhydrate. The flakes dissolve rapidly, leaving no sediment.

For a sample, write us today on your business letterhead.

Want insurance against blue blotches? Iron in your unhairing solutions can cause blue blotches in your leather.

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Available in 600-lb. drums of 40% solution.

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- ☐ 502 Unhairing Hides and Skins
- ☐ 503 Studies in Unhairing
- ☐ 505 Sodium Tetrasulfide for the Leather Industry

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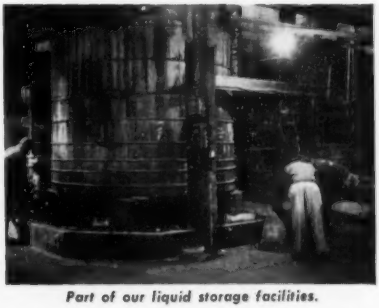
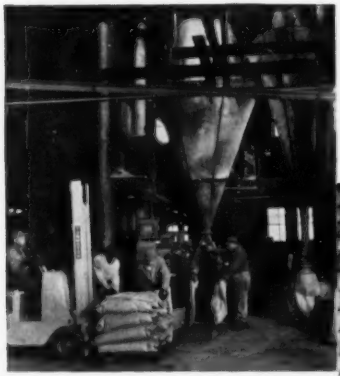
RIVER PLATE CUSTOMERS have Product as well as Service Dependability



Part of our liquid storage facilities.



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SOLIDOGEN	Dyefixing agent to improve wash fastness of color.
CHEELOX NULLAPONS	Sequestrants to prevent dulling and change of shade.

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BLANCOL	Syntan for bleaching light leather.
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NEUTRALIZING

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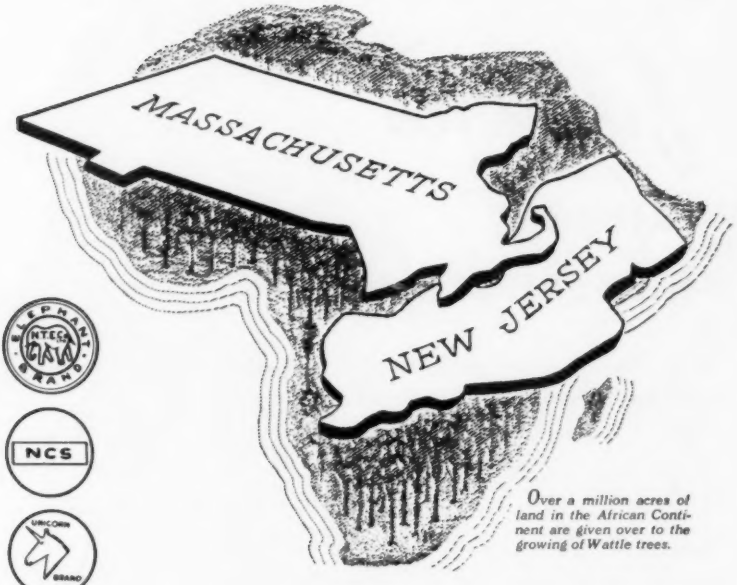

Over a million acres of land in the African Continent are given over to the growing of Wattle trees.

How big is 1,000,000 acres? It's more than the combined areas of two states prominently identified with leather production . . . Massachusetts and New Jersey.

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Wattle is a pure extract, untreated in any way by chemicals. It keeps the salts content of tanning liquors down to a minimum. Clear solutions, regardless of pH, with least sludge deposits, are the inevitable result.

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Every industry has its famous names. In tanning, Atlas, and the products it has developed through the years, all are "household names"—respected because they represent the finest of their kind and for their purposes.

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Atlas, keeping apace of, and frequently leading the way to, new progress in tanning, brings you the products best suited to your needs.

It is a fact that when tanners change to Atlas Fatliquors, few ever change again.

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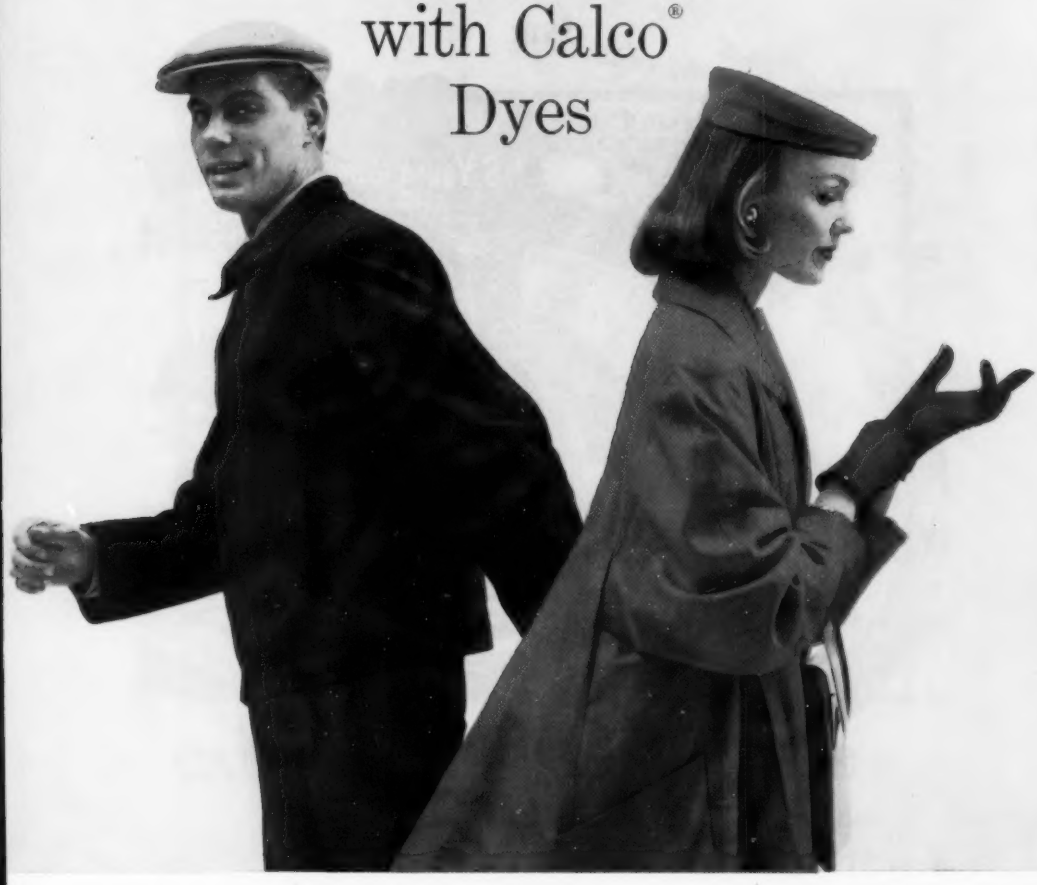

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Calcofast* Dyes

For a full range of pastel and medium shades on all types of leathers, specify Calcofast Dyes. They're tops for lightfastness, levelness, clarity and stability.

Your Cyanamid Dyes representative will gladly supply samples and full information on the following Calcofast Dyes for leather or other dyes in the extensive Cyanamid line.

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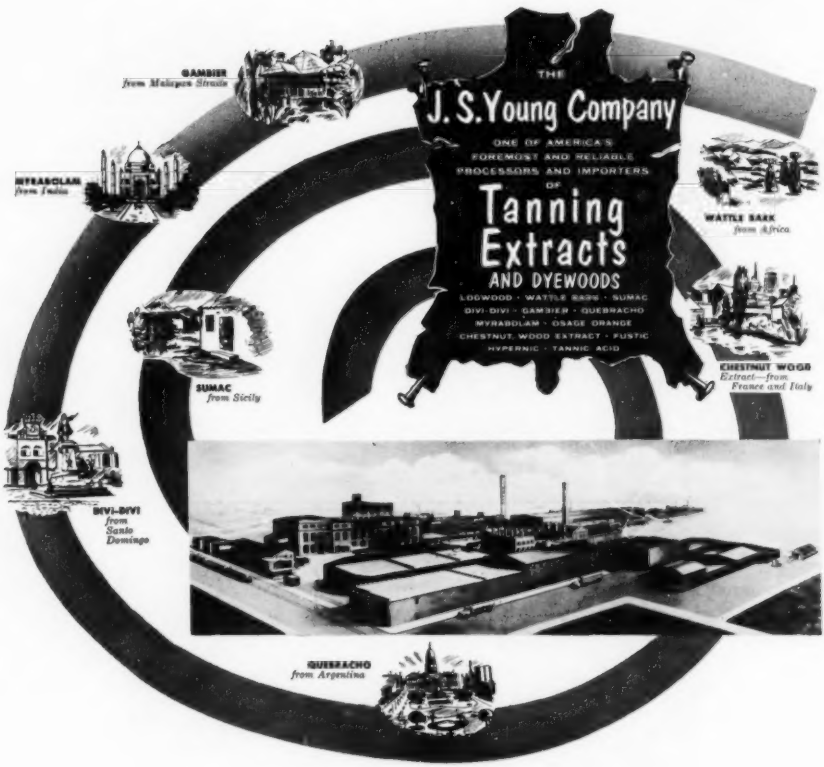
Symbol for Quality Dyes



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Our skilled technicians and research facilities are available to help you meet your individual problems . . . to meet your specifications and requirements.

There's no substitute for experience . . . no alternate for the kind of facilities provided by the J. S. YOUNG COMPANY.

Our firm has the reputation, over all these years, for prompt delivery direct from Baltimore, or from our warehouses in Peabody, Mass., and Chicago . . . shipments in any quantity—from barrel to tank car or tank truck.

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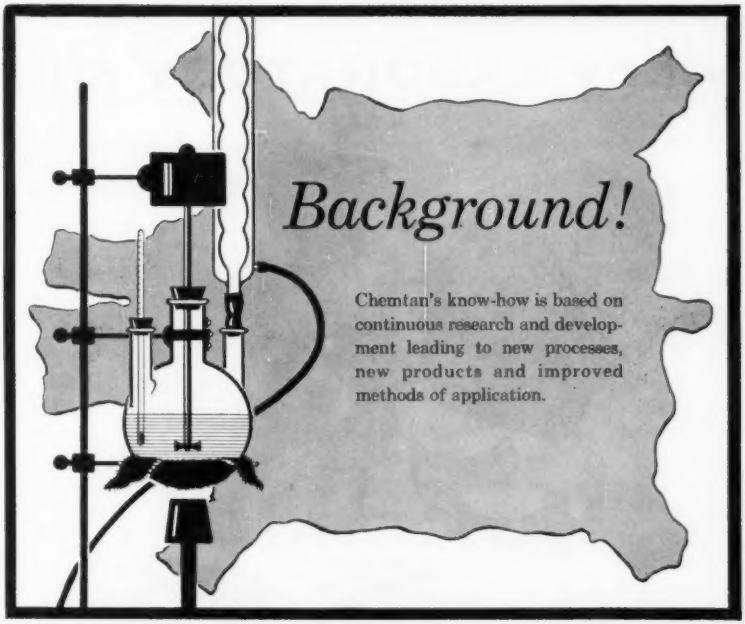
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Chemtan's know-how is based on continuous research and development leading to new processes, new products and improved methods of application.

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with Trojan Powder Company's pure Calcium Formate. Some of the advantages are:

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Uniformity and speed of tannage
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Lower production cost

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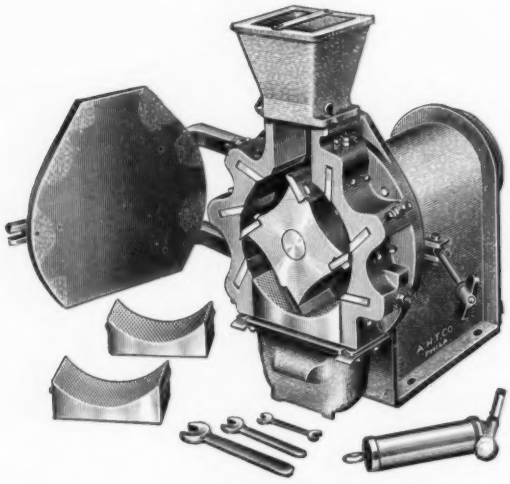
All our facilities are devoted exclusively to the tanning industry

New Standard Model No. 3 . . .

WILEY LABORATORY MILL



4274-V.

Wider range of use . . . Quieter operation . . . Increased safety . . .

For preparation, with minimal loss of moisture from heating, of wide variety of materials for analysis. Principal advantages of new model: harder cutting edges permitting wider range of materials, including Teflon, polyethylene resins, etc.; quieter operation; provisions for greater safety; and Hammertone gray enamel and chromium plated finish.

As in earlier model, four hardened steel knives on revolving shaft work with shearing action against six knives bolted into frame. Shearing action of cutting edges, between which there is always clearance, minimizes loss of moisture, avoids temperature rise, liquefaction, contamination, etc., making this mill satisfactory for many materials which cannot be reduced by other mechanical means. Ground material must pass through a sieve dovetailed into frame.

With either cast aluminum drawer, 28 oz. capacity, or interchangeable chute for collecting sample directly in a standard jar, 16 oz. capacity. Mill without motor or base is 21 inches high, occupies table space 14½ x 19 inches. Motor driven model is mounted on enclosed base 16¼ inches high.

4274-T. Wiley Laboratory Mill, Standard Model No. 3, with chute, three 1-pint jars, three sieves of 0.5 mm, 1 mm and 2 mm mesh, respectively, pulley for V-belt, set of wrenches, and grease gun, but without motor 576.50

4274-V. Ditto, but with drawer in place of chute 567.00

NOTE—Both models also furnished complete with ½ h.p. motor with starting switch and thermal overload cutout, for 115 volts, 60 cycles, single phase a.c., mounted on enclosed base, at $886.00 and $876.50, respectively.

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PENETRATION OF WATTLE TANNINS INTO HIDE AS EXAMINED BY CHROMATOGRAPHIC AND MOLECULAR WEIGHT METHODS

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Grahamstown, South Africa

ABSTRACT

Chromatographic and molecular weight studies were made on each member of a set of wattle liquors used for tanning sole leather. There is an accumulation of the low RF, higher molecular weight tannins in going from the head (fresh) to the tail (spent) end of the set, due presumably to faster penetration of the higher RF, lower molecular weight components.

INTRODUCTION

Penetration of tannins through the capillaries of the grain and flesh layers into those of the general hide structure is an important physical process which precedes tannin fixation.

The speed of penetration depends on a number of variables in both liquors and hides, e. g., temperature, viscosity, pH value, and salt content of the extract; degree of swelling of the hide; and the agitation of the liquor or the hide (1). The condition of "case hardening", in which the tannins cannot penetrate into the central hide structure, and the common condition of "drawn" grain are well-known examples of arrested and retarded penetration.

Studies on comparative penetration rates have been made from time to time (2), but variable results have thus far been obtained due to the large number of factors involved. Profitable extension of this work has long awaited more selective methods of examining vegetable extracts and the development of a dependable method of molecular weight estimation over the range 500–5,000. In this study such techniques are used to examine the changes in composition and average molecular weight of the tannins remaining in the liquors during the course of tannage.

CONDITIONS OF TANNAGE

The pilot plant sole leather tanning process, examined by chromatographic and molecular weight methods, was operated under the following conditions:

Complete tannage was effected in an eight-pit pressover system in which 100°Bk. black wattle (*Acacia mollissima*) liquors containing ½% salt were introduced daily (6 times per week) into the top pit. At the spent end, limed steer hide pieces, pickled for 24 hours in a solution containing 5% sodium chloride by volume and 2% sulfuric acid on the limed stock weight, were also introduced daily (6 times per week). The fresh (100° Bk.) liquors introduced were adjusted to pH 3.3, and all liquors were readjusted to this pH daily if necessary.

EXAMINATION OF THE SOLE LEATHER PROCESS

Chromatrography.—Chromatography was done substantially as described in an earlier paper (6). The liquors from each pit were spotted on to two-dimensional paper chromatographic sheets, without prior purification. The weaker liquors from the spent end were concentrated slightly under nitrogen at reduced pressure merely to facilitate spotting on.

The chromatograms were irrigated with water-saturated sec.-butanol in direction 1 and water only in direction 2, and sprayed with ammoniacal silver nitrate. (Figs. 1–8).

The spent liquor (Fig. 8) was freed of gums by alcohol precipitation (3) and similarly examined (Fig. 9).

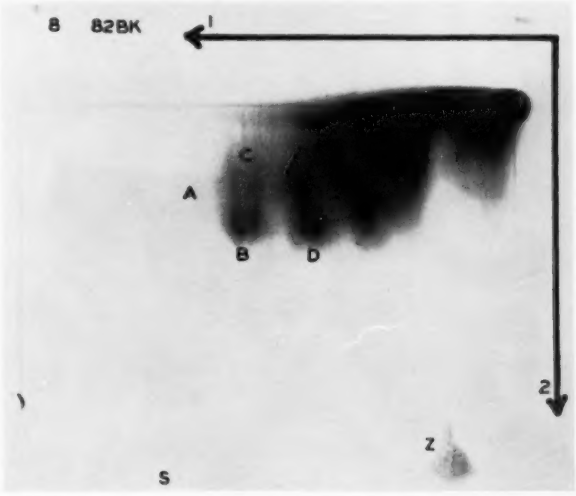

FIGURE 1.—Two-dimensional chromatogram of sole-leather tannage (concentrated end). Pit No. 8

Direction 1 Water-saturated sec.-butanol
Direction 2 Water only
S denotes solvent front in direction 2.
Bk values indicated are those of the liquors.

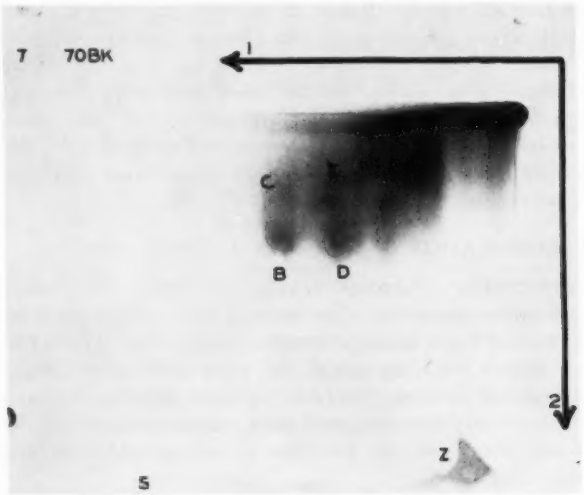

FIGURE 2.—Sole-leather tannage. Pit No. 7. See Fig. 1 for legend.

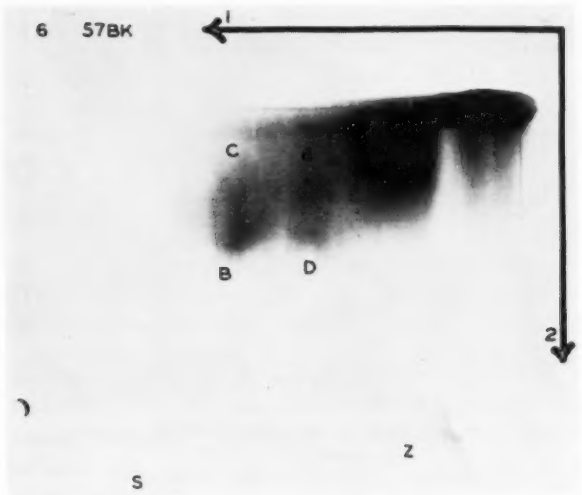

FIGURE 3.—Sole-leather tannage. Pit No. 6. See Fig. 1 for legend.

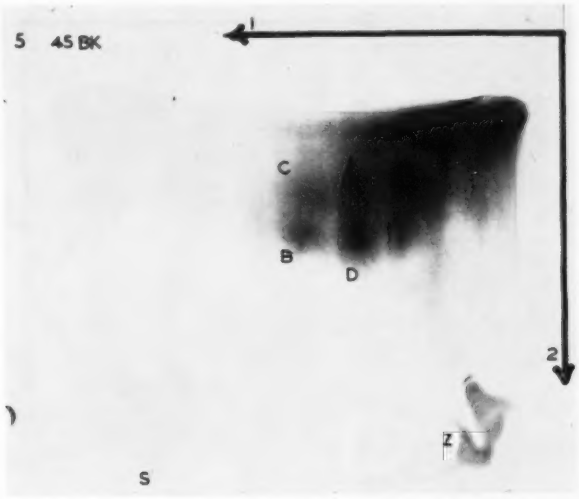

FIGURE 4.—Sole-leather tannage. Pit No. 5. See Fig. 1 for legend.

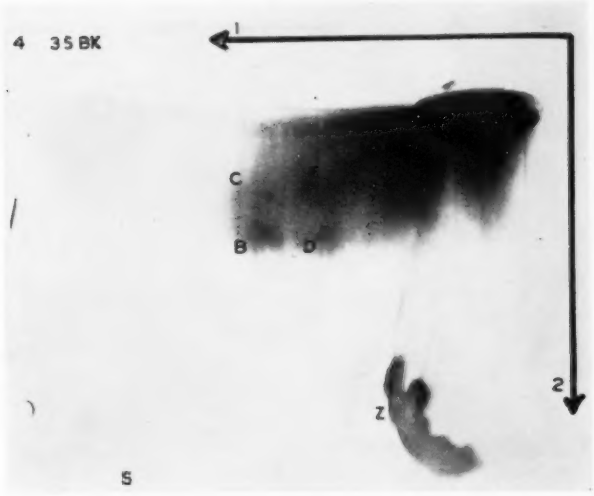

FIGURE 5.—Sole-leather tannage. Pit No. 4. See Fig. 1 for legend.

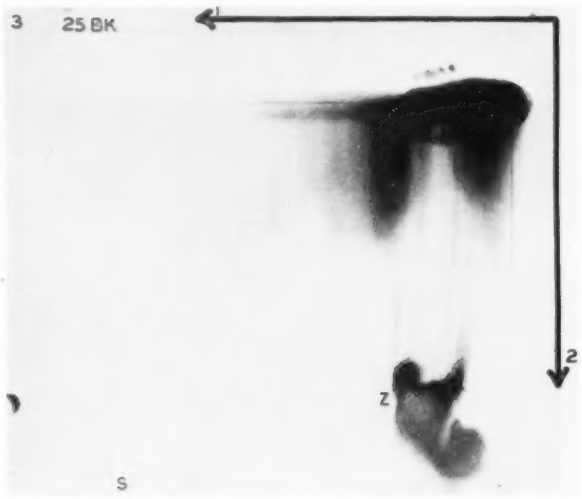

FIGURE 6.—Sole-leather tannage. Pit No. 3. See Fig. 1 for legend.

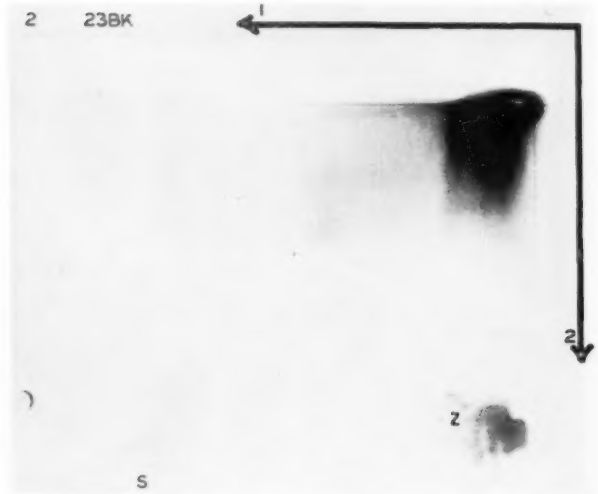

FIGURE 7.—Sole-leather tannage. Pit No. 2. See Fig. 1 for legend.

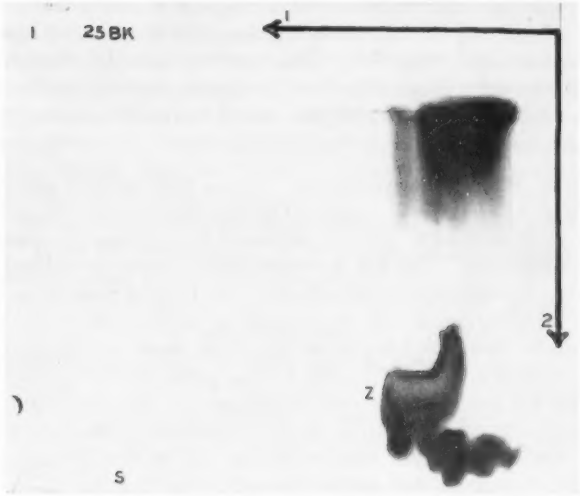

FIGURE 8.—Sole-leather tannage (spent end). Pit No. 1. See Fig. 1 for legend.

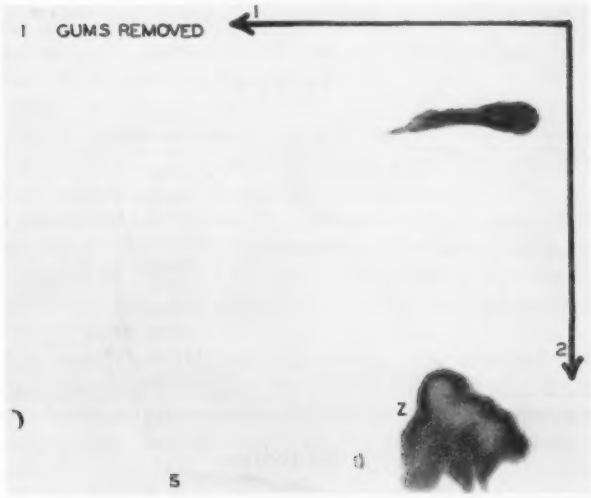

FIGURE 9.—Tannins in spent liquor after removal of gums. See Fig. 1 for legend.

A large gray area, *Z*, which moves just behind the solvent front in direction 2, is evident on all the chromatograms and increases in concentration toward the spent end (Figs. 5–9). This "spot", which develops on spraying, is due to the presence of salt (NaCl) in the liquors, introduced mainly through the pickled hide at the spent end and also in low concentration (½%) from the concentrated end.

Molecular Weight Estimations.—Liquor from each pit was evaporated to dryness in a current of nitrogen. The dry solid residue was exhaustively extracted with methanol, and the methanol extract was evaporated under the same conditions. The dry methanol extract was methylated with diazomethane (5) which simultaneously frees the tannins from sugars and gums. Complete methylation of the polyphenols or tannins, which is an essential prerequisite for their solubility in benzene (the most suitable solvent for determining molecular weights by the boiling point method), was difficult to effect. This was partly due to introduction into the liquors of impurities such as acids and salts which catalyze the decomposition of diazomethane. This difficulty was overcome by readjusting the pH of the liquors to 4.6 (the natural pH of concentrated wattle extract) before the initial concentration under vacuum, and also by carrying out the methylation reaction at temperatures below 0°C., for 3 to 7 days, under rigidly applied freezing conditions. A sufficiently high degree of substitution was ensured after two methylations with this experimental technique.

Molecular weights of the methylated tannins were determined with the modified Ray ebulliometric apparatus (4). The results obtained are given in Table I.

TABLE I

Pit No.	Molecular Weight
Fresh extract	1267± 2
8	1146± 2
7	1225± 8
6	1373± 13
5	1342± 4
4	1301± 5
3	1435± 18
2	1640± 23
1	1478± 19*
Spent liquors	1325± 6*

*Values subject to confirmation. Obtained by successive methylation and acetylation of the tannins.

DISCUSSION

An examination of the chromatograms of pits 8, 7, 6, 5, and 4 (Figs. 1–5) shows that, although no spectacular changes take place in these pits during tan-

nage, a slow decrease in the proportion of the mobile constituents (*A*, *B*, *C*, *D*, and *E*) and a corresponding increase in the proportion of low RF tannins occur. This is especially noticeable when comparing pit 4 (Fig. 5) with pit 8 (Fig. 1). Over these pits a gradual increase in the average molecular weight occurs (see Table I). As the mobile constituents are known to be of small molecular weight (about 620 [7]), both the chromatographic and molecular weight evidence show that there is a preferential penetration of the partly tanned hide by the "nonastringent" molecules of the concentrated liquors. These "nonastringent" small molecules, which yield spots *A*, *B*, *C*, *D*, and *E*, are known to have a lower affinity for collagen than the larger molecules which tend to remain in the liquors (6).

Comparison of pit 4 (Fig. 4) with pit 3 (Fig. 5) shows a spectacular reduction in the proportion of mobile constituents *A* to *E*; only a mobile constituent (direction 2), of lower RF than *A*–*E* in direction 1, remains. This is accompanied by a marked rise in molecular weight to 1435 (Table I). In the next pit (No. 2) this constituent is also mainly absorbed by the hides, and the residual tannins in the liquor increase in average molecular weight by 205 units to 1640.

Contrary to existing tanning theory, therefore, small molecules with relatively low affinity for collagen (6) are preferentially absorbed from the stronger liquors, probably due to their more rapid diffusion into the hide structure. This effect is emphasized even more toward the weaker end, and the final spent liquor contains "astringent" tannins of high affinity for collagen. Molecular weight "screening" occurs during the penetration of wattle tannins into hides, and molecular size (as distinct from molecular weight) appears to be a critical factor involved in this process. The practical significance of these results is discussed by Shuttleworth (8).

The presence of tannins with a high affinity for cellulose, and therefore collagen (6), in "mellowed" spent liquors is an apparent inconsistency which is under investigation. Two explanations are possible for this behavior:

(*a*) A low percentage of "astringent" tannins appears to be adsorbed on the gums present in spent liquors (3). The gums may be partly, but not completely, detanned with 90–95% ethanol (3). The strong adsorption of "astringent" tannins by the gums will modify their affinity for collagen and also inhibit their penetration into the hide due to the very high molecular weight and size of the gums.

(*b*) Other tannins of large molecular weight, not adsorbed by the gums, may surface-tan the fresh hide introduced into pit 1. After surface-tannage further penetration of the high molecular weight tannins into the hide structure might be prevented. One or both of these factors may be involved.

Roux (6) has shown that the affinity of individual black wattle polyphenols for collagen varies enormously, and may be dependent on a combina-

tion of various physical and chemical factors, e. g., solubility in water, spatial orientation, molecular size, and number and position of functional groups. From the paper chromatographic and molecular weight evidence presented above there appears to be a direct relationship between affinity for cellulose (and collagen) and molecular weight of black wattle tannins.

More recent studies (9) of the molecular weight of the tannins in the spent liquors from experimental single-pit tannages also show substantial increases over those of the original liquors. These findings confirm the relatively more rapid penetration of the low molecular weight tannins with low affinity for collagen compared with those of high molecular weight.

SUMMARY

1. Paper chromatography and molecular weight estimations are used to study a pilot plant vegetable tannage of sole leather pieces with commercial black wattle extract.

2. Paper chromatograms of the contents of each pit show that during tannage the mobile constituents of low affinity for collagen are preferentially absorbed, probably due to their more rapid diffusion into the hide structure, leaving an immobile (low RF) "astringent" tannin fraction in the liquor toward the spent end.

3. Accurate molecular weight estimations show that the tannins remaining in the pits during tannage gradually increase in molecular weight from the concentrated to the spent end. A large increase in molecular weight of the tannins occurs toward the last liquors at the spent end.

4. This evidence, which is contrary to existing tanning theory, shows that a certain amount of screening based on molecular size occurs during tannage with black wattle extract.

5. A direct relationship between the affinity of black wattle tannins (for collagen and cellulose) and their molecular weight appears to exist.

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SPECTROPHOTOMETRIC STUDIES, PART VI CHROMIUM COMPLEXES

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ABSTRACT

Spectrophotometric curves in the ultraviolet region have been obtained for basic chromium sulfate solutions complexed with various organic acids and mixtures of these acids and their salts. The mode of complex formation has been investigated both by molar ratio method and Job's method of continuous variations. The results have shown that the methods are applicable in several cases to the elucidation of these coordination compounds. The nature of the chromium complexes is discussed in the light of these results.

INTRODUCTION

Theis *et al.* (1–6), as a result of spectrophotometric studies in the visible region on chrome liquors containing different amounts of various organic acids, grouped the acids according to their ability to penetrate into the chromium complex.

Serfass, Theis, and Clark (1–3) found that the absorbance due to oxalatochromiate complexes increased to a constant maximum value as the proportion of oxalate to chromium was increased. By comparing this maximum absorbance with the absorbance obtained when Cr and oxalate were present in the stoichiometric proportions of trioxalatochromiate, the percentage of penetration of oxalate into the chromium complex was calculated. It was assumed that only the trioxalatochromiate complex was formed in solution. No attempt was, however, made to find the ratio of the reacting constituents in the complex from the data.

Shuttleworth (7–12), from conductometric studies, postulated certain formulas for the complexes of chromium with a number of anions. Küntzel (13,14) also studied the problem in detail, and presented schematic formulas for several chrome complexes. Green (15) recorded data for the chromium-alanine complex.

Previous spectrophotometric studies all pertain to the absorbance of the chromium complexes in the visible region. In the present study, the absorbance characteristics of chromium complexes in the ultraviolet region have been measured, since the chelate and inner complexes may have better characteristic absorption in the ultraviolet than in the visible region.

Several methods (16) have been suggested for the spectrophotometric study of complex formation. In the molar ratio method (17), a plot of the optical density against the molar ratio of component *B* to component *A*, where *A* is constant, gives a straight line rising from the origin and breaking sharply to constant optical density at the molar ratio of the components in the complex, provided the complex is quite stable. If the complex undergoes dissociation, constant optical density is achieved only when an excess of the variable component is added. In Job's method of Continuous Variation (18), solutions of *A* and *B* (where *A* is a metallic ion and *B* is a molecule like NH_3 or an anion) of the same molar concentration are mixed in varying proportions, and a suitable property like absorbance is measured. The difference between each absorbance value found and the corresponding value calculated assuming no reaction, is plotted against the composition. The resulting curve exhibits a maximum or minimum corresponding to the molar ratio of components in the complex. The maximum bears a simple relation to the number of moles of *B* reacting with one mole of *A*, and is independent of the equilibrium constant, i.e., the degree of dissociation of the complex.

The foregoing methods have been applied to the study of basic chromium sulfate complexes with oxalic, tartaric, citric, aminoacetic, chloroacetic, glycolic, formic, and acetic acids and their salts, and the results are reported in this paper. Structural formulas for the complexes formed are suggested.

EXPERIMENTAL

Materials.—33⅓% basic, chromium sulfate liquor (free from neutral salts) was prepared (courtesy of Mr. P. S. Venkatachalam) by reducing reagent-grade chromic acid with sulfur dioxide and adding the calculated amount of barium hydroxide slurry. The precipitated barium sulfate was filtered off and the liquor aged for two months. The chromium content as Cr and basicity of the liquor, determined by standard methods, proved to be 52.02 g/l and 32.47% respectively. Solutions of standard molarities of organic acids and salts were prepared by weighing the required amounts of pure reagent chemicals and dissolving them in calculated quantities of distilled water.

Methods.—For the study by the molar ratio method, 1 ml. of 1 *M* Cr solution was mixed with various quantities of 1 *M* acids, e.g., 0.5, 1, 2, 5, and 10 ml. of 1 *M* oxalic acid, to give molar ratios of organic acid to Cr of 0.5,

1, 2, 5, and 10 respectively. The solutions were diluted to the same volume (11 ml.) and allowed to age for one month.

In the continuous variation method, solutions of chrome liquor and the organic acid having the same molarities were prepared, and a number of solutions with varying ratios of the two components were made by adding x ml. of organic acid solution to $1-x$ ml. of chrome solution.

The absorbance readings were obtained with a Beckmann D. U. Spectrophotometer using the ultraviolet accessory set with a 1-cm. quartz cell. The solutions were suitably diluted to give measurable absorbance readings. The wave length range studied was the near ultraviolet region, 230–350 mμ.

Since the Cr solutions themselves absorb ultraviolet light, their absorbance readings together with those of organic acids and salts which absorb ultraviolet light were determined at all the concentrations employed. The acids, excepting glycolic acid, had little absorbance in the range 240–350 mμ. The absorbance of the test solution was measured, and after deducting the absorbance due to the chrome and acid or salt, if any, for the respective concentrations, the differential absorbance readings were plotted against moles of organic acid per mole Cr in one method, and against the ratio of organic acid or salt to Cr in the other method.

RESULTS

Molar Ratio Method.—The data obtained for optical density when increasing quantities of different acids are added to one mole of Cr are given in Figs. 1–10. From the data, the acids may be grouped into four categories:

I. HCl and H_2SO_4. — The absorbance values decrease linearly in the initial stages (up to 2 moles addition in the case of HCl and 5 moles addition in the case of H_2SO_4) and then attain a constant value for molar ratios of 1:5 to 1:10.

II. Acetic, formic, and chloroacetic acids. — Absorbance values decrease linearly in the initial stages, attaining a minimum at ½ – 1 mole acetic, 2 moles of formic acid, and 1 mole of chloroacetic acid per mole Cr, after which the values tend to increase linearly. This behavior is most pronounced in the region of 235–245 mμ.

III. Glycolic, tartaric, citric, and oxalic acids. — With these acids the absorbance values increase with the increase in the amount of acid added; a definite break in the curve is observed in each case at 2 moles of acid per mole of Cr.

IV. Aminoacetic acid. — The absorbance values increase linearly to a maximum at 1 mole acid addition, and the values then decrease drastically and linearly with further increase in the amount of acid added. Negative values are obtained at 5–10 mole additions.

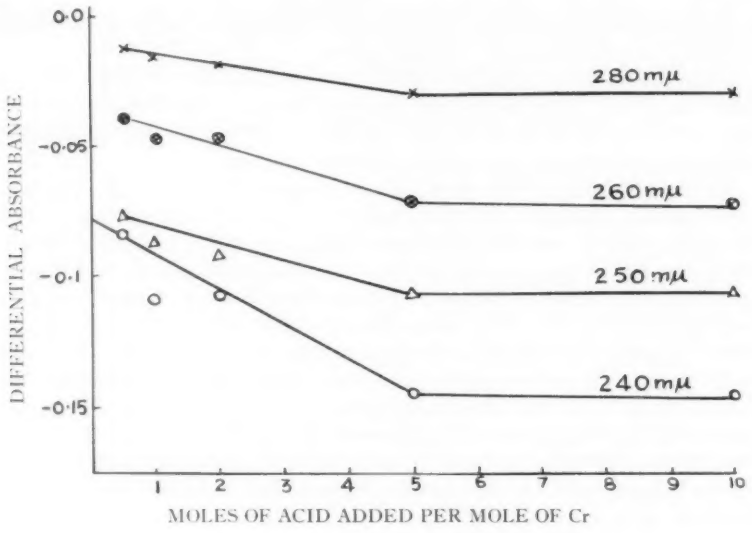


FIGURE 1.—Absorbance of chrome liquors treated with sulfuric acid; molar ratio method.

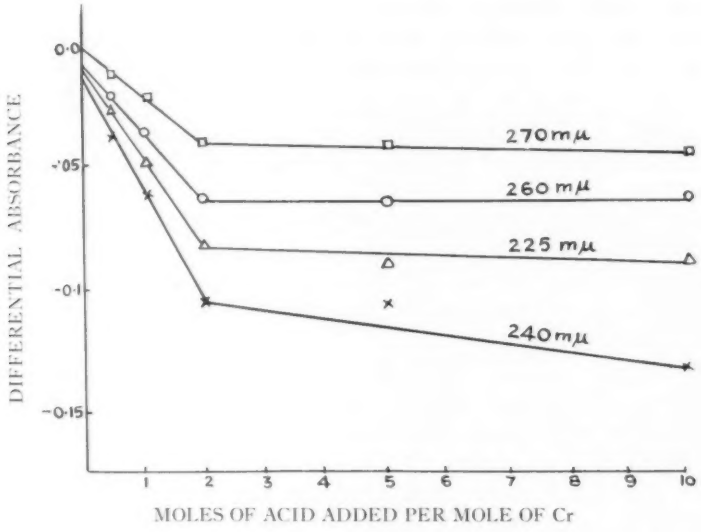


FIGURE 2.—Absorbance of chrome liquors treated with hydrochloric acid; molar ratio method.

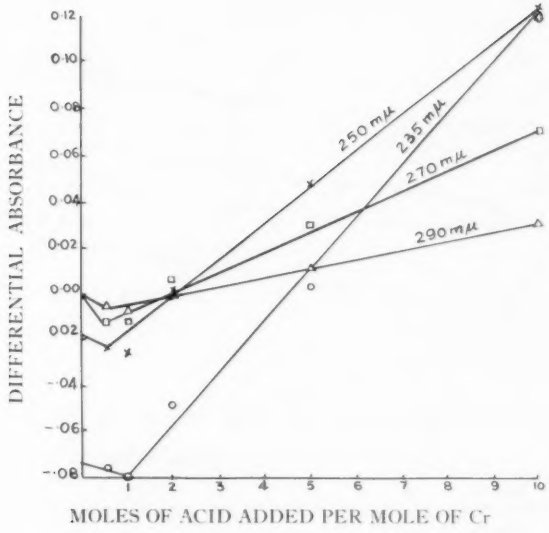


FIGURE 3.—Absorbance of chrome liquors treated with acetic acid; molar ratio method.

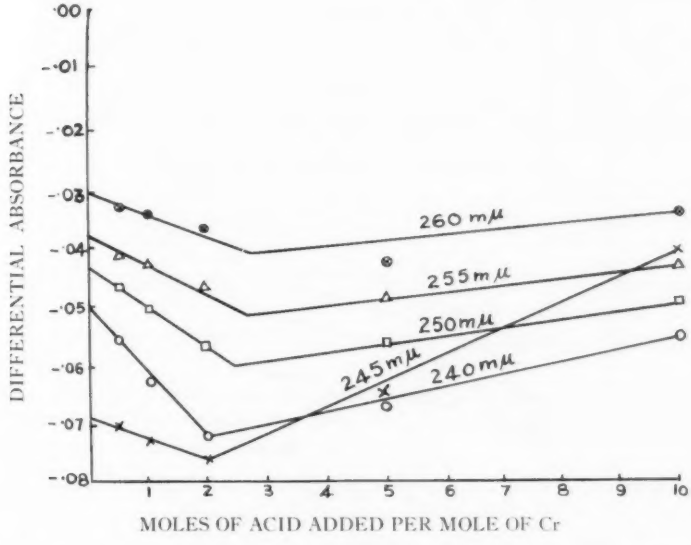


FIGURE 4.—Absorbance of chrome liquors treated with formic acid; molar ratio method.

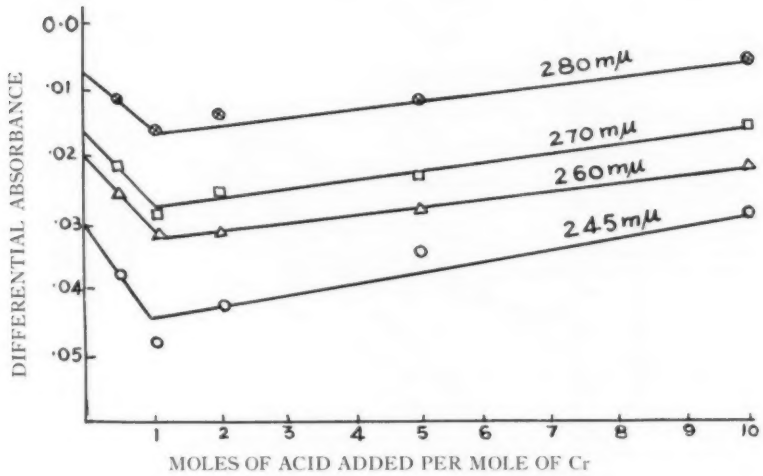


FIGURE 5.—Absorbance of chrome liquors treated with chloroacetic acid; molar ratio method.

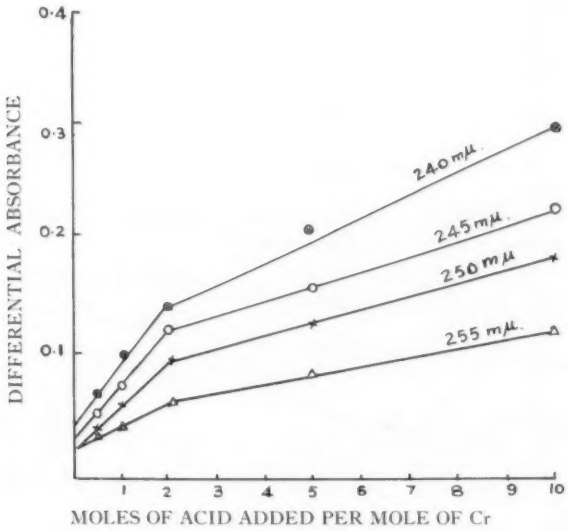


FIGURE 6.—Absorbance of chrome liquors treated with glycolic acid; molar ratio method.

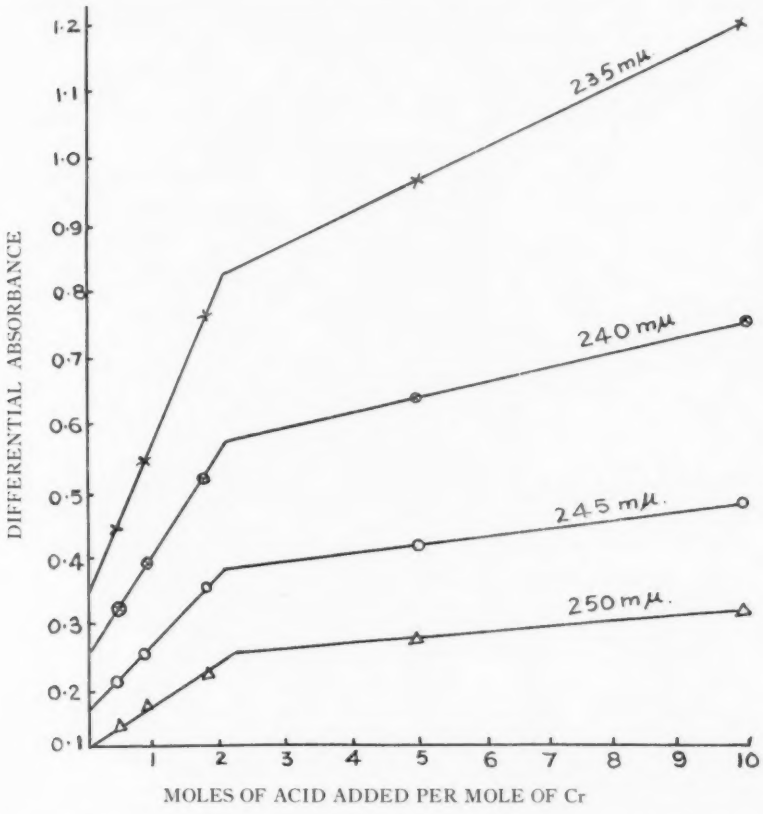


FIGURE 7.—Absorbance of chrome liquors treated with tartaric acid; molar ratio method.

The various factors that are involved in the addition of an acid to chrome liquor are: (*a*) lowering of the pH of the liquor, (*b*) hydrolysis of the basic chromium sulfate, (*c*) deolation, (*d*) breaking up of aggregates, and (*e*) complex formation.

Complex formation may take place either with basic Cr compounds or with the compounds which have been deolated or otherwise modified by the presence of acid and decrease of pH.

Another important point is that the absorbance readings for the blank are those of the basic chromium solution at a certain pH. With the addition of various quantities of acid, the pH is reduced to varying extents. It is known that the absorbance readings of chromium liquor vary with pH. Though the

data for chromium liquor at various pH values were obtained (by adjustment with H_2SO_4), the values could not be used to make the necessary corrections since complications arose due to deolation and other factors. This is perhaps the reason why the curves do not pass through the origin. From the foregoing observations, it is readily seen that the curves cannot be interpreted quantitatively. However, the following interpretation of a qualitative nature was possible and formed the basis of further work by the other method.

The sulfuric and hydrochloric acids deolated the basic chromium sulfate complex, complete deolation taking place at 5–10 mole ratios of acid to chromium.

The chloroacetic acid, formic acid, and acetic acid either caused partial deolation, with complex formation taking place slowly with the deolated complex, or the acids formed unstable complexes which are characterized by the minima.

The oxalic, tartaric, glycolic, and citric acids showed complex formation corresponding to 2:1 molar ratio of the acid to Cr, since a characteristic break was observed at this point. However, the absorbance readings did not reach constant values even by the addition of 10 moles of acid. This is in accordance with the data obtained in the visible region by Theis *et al* (1–6). These results indicate that the complex undergoes appreciable dissociation in solution. It is interesting to note that the break occurs at the same point for all four acids. However, in the case of the acids containing more than one carboxyl group, it is plausible to assume that they coordinate only through their ionized groups, depending on the pH of the solution and the ionization of the respective groups. Structural formulas could be predicted, employing 2 moles of acid for 1 Cr in each case, but it is necessary to know whether the basic chromium sulfate undergoes deolation or not due to the decrease in pH. Some deolation ought to take place, since the complexly bound sulfate is displaced by the penetrating anion of the acid, which in turn should have an effect on the basic chromium complex. Unfortunately, the basicities of the solutions could not be determined since the Lehigh method is not applicable in systems containing weak acids (19). The effect of organic acid on the hydrolytic equilibrium of the basic chromium sulfate and on aggregate formation is not precisely known, which causes further difficulties in picturing the structural formulas of the complexes.

With aminoacetic acid there is a maximum at 1 mole of added acid per mole of Cr by the molar ratio method. The maximum may be caused by the decrease in pH at higher mole ratios. Further, the pH of the aminoacetic acid itself was 5.5, which may have adversely affected the differential values. However, the break is very significant and may represent the nature of the complex empirically.

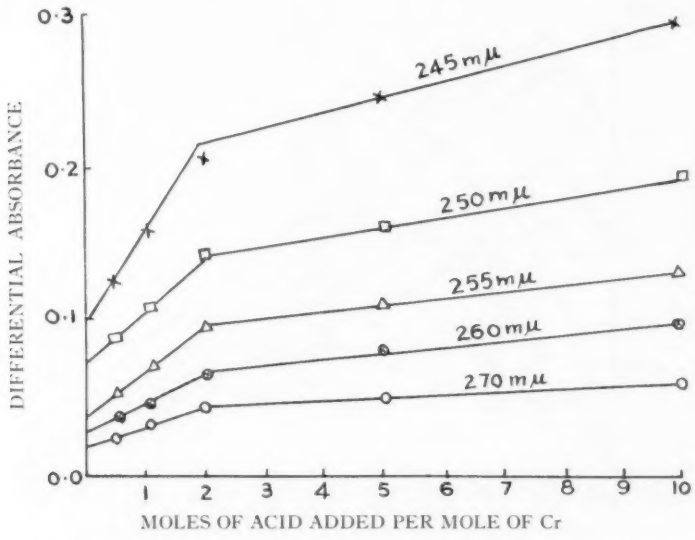


FIGURE 8.—Absorbance of chrome liquors treated with citric acid; molar ratio method.

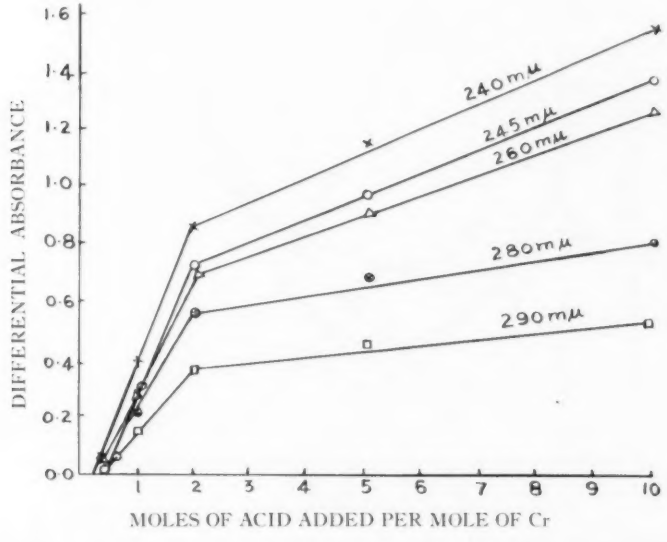


FIGURE 9.—Absorbance of chrome liquors treated with oxalic acid; molar ratio method.

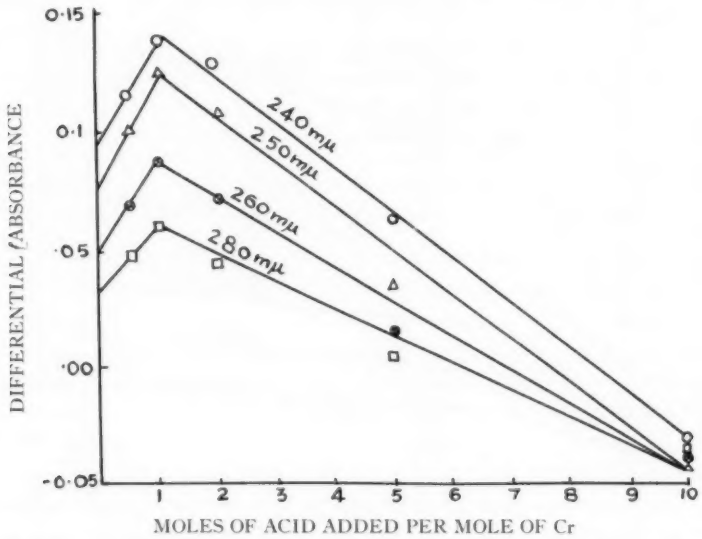


FIGURE 10.—Absorbance of chrome liquors treated with aminoacetic acid (glycine); molar ratio method.

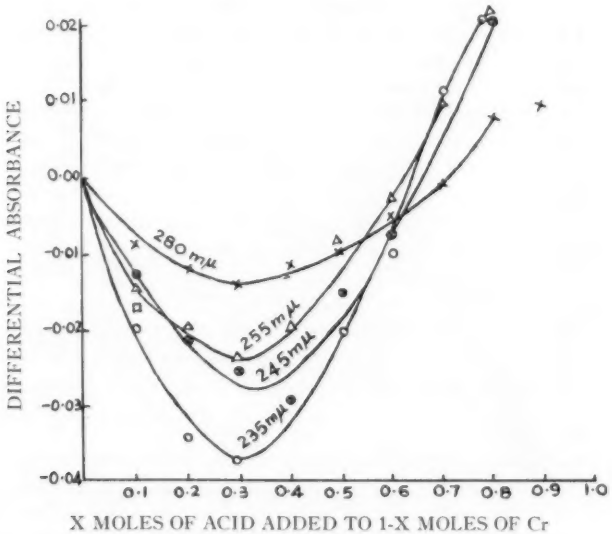


FIGURE 11.—Absorbance of chrome liquors treated with acetic acid; continuous variation method.

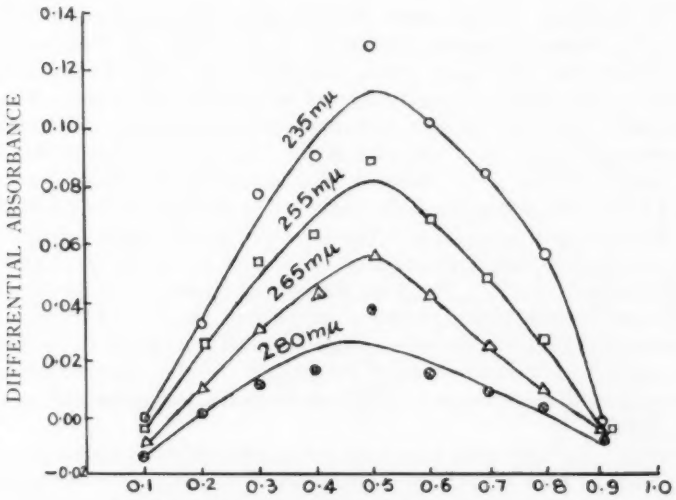


FIGURE 12.—Absorbance of chrome liquors treated with aminoacetic acid (glycine); continuous variation method.

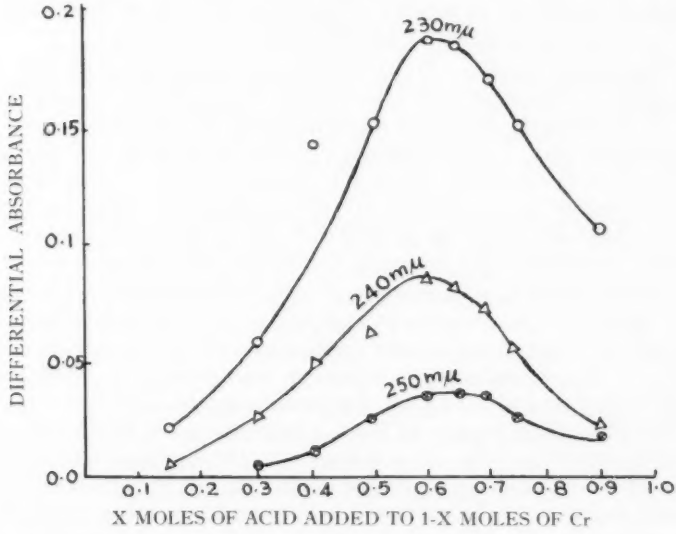


FIGURE 13.—Absorbance of chrome liquors treated with glycolic acid; continuous variation method.

Job's Method (Continuous Variations).—The application of Job's method to chromium liquors with added organic acids and their salts has given results which are more specific and significant. Figs. 11–14 give the curves for acetic, aminoacetic, glycolic, and oxalic acids. The results obtained by the molar ratio method have been practically confirmed by this method, since complex formation is indicated at the same molar ratios in this case also, namely, 1:2 for acetic (denoted by a minimum), 1:1 for aminoacetic, and 2:1 for glycolic and oxalic acids. However, in the case of oxalic acid there is an inflection corresponding to 3:1 in the wave length region 265–290 mμ. Hence the simultaneous formation of two complexes is indicated even at low pH values (1.5–2.0). The data, though significant, could not be interpreted quantitatively since the results are vitiated again by changes in pH. It is quite probable that the molar ratios obtained correspond to the nature of the complexes formed at the pH of the solution (1.8–2.4). Further evidence on the extent of deolation and basicity is necessary to confirm the validity of the results.

To obviate the difficulties caused by changes in pH and consequent deolation, acids and their salts of the same concentration (1 *M*) were mixed in such a way as to give slightly higher pH values (3.8–4.0) than that of the chrome liquor itself (3.1–3.2). It is necessary to keep the pH slightly higher so that the released sulfuric acid has little effect on the chromium complex and in turn is acted upon by the salt of the organic acid. Too high a pH of the mixture should also be avoided since in such cases further complications arise due to the displacement of the OH group.

In these instances also, though the curves exhibited pronounced and characteristic maxima, the pH value varied, though not to a great extent (3.8–2.5), owing to the changes of concentration of the chrome liquor and the acids and their salts. It was necessary, therefore, to adjust the pH of the individual solutions containing different combinations of Cr and anion to the pH of the chrome liquor (3.1–3.2) by adding very dilute sulfuric acid and NaON at intervals during a week, until there was no longer any change. The solutions were boiled in the beginning to obtain equilibrium quickly.

The results obtained for various organic acids and their salts by adjustment to constant pH 3.1–3.2 are given in Figs. 15–19. In spite of the precautions taken, no curves permitting definite conclusions could be obtained for acetic, formic, and chloroacetic acids in admixture with their salts. With acetic acid maintained at a pH of 3.1, no characteristic maxima could be obtained. However, after aging for over 15 days, a minimum could be detected at a ratio of 2 acetate to 1 Cr. This is similar to the minimum formed when acetic acid alone is added to the chrome liquor (Fig. 3). The formation of this minimum may probably be attributed to hydrolysis, which has already been mentioned as one of the factors affecting the studies on complex formation. The effect is exhibited increasingly when the chrome liquor is aged in the

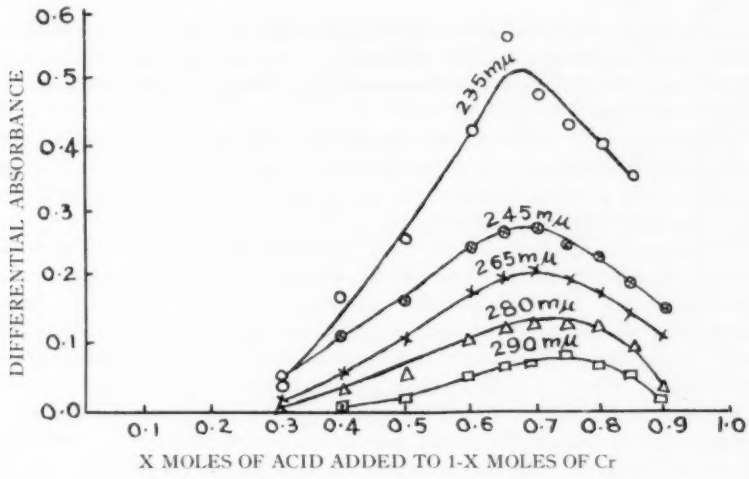


FIGURE 14.—Absorbance of chrome liquors treated with oxalic acid; continuous variation method.

presence of low concentrations of organic salt. Though the pH is adjusted, the hydrolysis is not checked because high dilutions are employed in the studies (.02–0.1 *M* Cr) and the alkali added neutralizes mainly the free mineral acid which is formed, either due to hydrolysis or to the displacement of the sulfate. The hydrolytic effects are reflected by the steady increase in the absorbance readings, especially at 235–250 mμ, without becoming constant. Of course the basicity ought to have increased, but difficulties encountered in the determinations of basicities of such liquors have already been referred to.

Whether added acetate had formed a complex at all was examined further by the differential method (20). A basic chromium sulfate solution, completely deolated by adding an excess of sulfuric acid and boiling, was taken as the blank. The absorbance of this solution was determined at wave lengths from 230 to 300 mμ. Absorbances were then measured at the same wave lengths for solutions of basic chromium sulfate alone, and for basic chromium sulfate plus acetic acid, at the pH value resulting from adding the acid, and at pH 3.2. All the solutions contained the same Cr concentration. The differential absorption for each solution at each wave length was obtained by subtracting the absorbance of the blank from the absorbance of the solution. These differential absorbances were plotted against wave length. The resulting curves showed maxima near 238 mμ for the basic chromium sulfate, at 248 mμ for the solution treated with acetic acid without pH adjustment, and at 253 mμ for the solution treated with acetic acid and brought to pH 3.2.

The curves for the latter two solutions are shown in Fig. 20. This shifting of the wave length at which the differential absorbance is maximum may indicate some complex formation between basic chromium sulfate and acetic acid or acetate.

In contrast with the data for acetic acid, the absorbance readings for formic and chloroacetic acids adjusted to pH 3.1 showed maxima at compositions of 1 acid to 1 Cr. However, the curves could not be drawn smoothly to give significant maxima, probably due to hydrolytic and similar effects. No quantitative interpretation of the data was therefore attempted. The minimum corresponding to that of acetic acid, though not well pronounced, is also obtained in the case of formic acid curves at that same composition, indicating that hydrolytic effects are probably vitiating the results. That the maxima may denote some complex formation with formic acid is also evident from the differential data which show a small maximum at 250 mμ for formic acid just like that of acetic acid. It is plausible that some unstable and nonchelate type of complex formation may take place in the case of these acids or their salts, but in order to predict their nature and the probable structural formulas, further pertinent and factual evidence is needed.

The continuous variation method has yielded precise data for the hydroxyacids. With glycolic acid-glycolate adjusted to pH 3.1–3.2 (Fig. 15) a well defined maximum is obtained at the 0.32–0.33 ratio corresponding to a

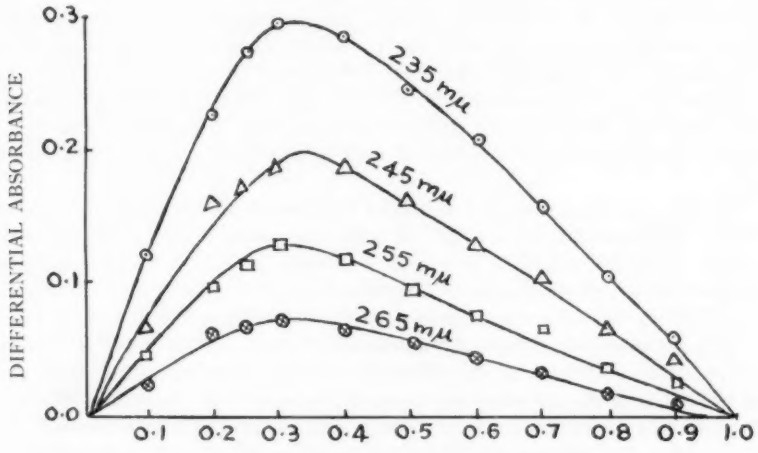
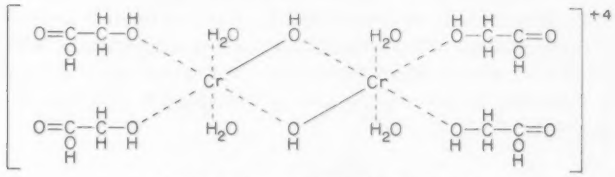


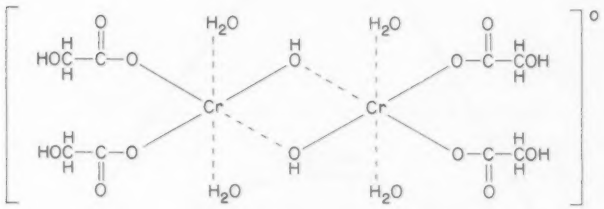
FIGURE 15.—Absorbance of chrome liquors treated with glycolic acid and sodium glycolate at pH 3.2; continuous variation method.

composition of 1 Cr to 2 glycolate molecules. In the case of the hydroxyacids, constant absorbance readings and pH values could be obtained within a week and changed little on subsequent aging, indicating that hydrolytic effects are not appreciable. Further, since the pH was the same as that of the original liquor, the olated OH groups probably are not disturbed. In such a case, it is very simple to predict possible formulas for the glycolate complex, namely:



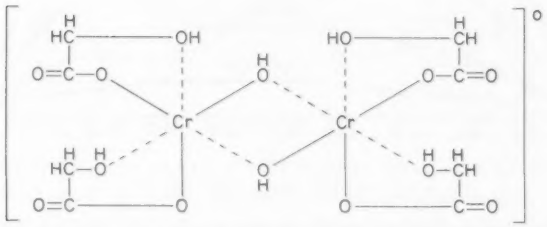
(I)

(Only the hydroxyl groups penetrate.)



(II)

(Only the carboxyl groups penetrate.)



(III)

(Both the carboxyl and hydroxyl groups penetrate, forming an inner chelate complex.)

Though indicating an anionic complex, Formula III is most likely because of the proximity of the OH and COOH groups, forming stable 5-membered chelate rings. The formation of such chelate complexes has been recognized in the inorganic field in general and in the leather field in particular. The higher absorbance values obtained with hydroxyacids and dicarboxylic acids like oxalic, compared to formic and acetic, point to a more stable form of combination as denoted by the chelate ring.

Some doubt may arise with regard to Formula III since it is anionic, with 2 negative charges per Cr atom, though the glycolate added is only in stoichiometric proportion. Theis *et al.* have observed (5) that the total cationic complexes decrease only gradually during the initial stages of addition of organic anion, and that only with a large excess of anion do the cationic complexes decrease to a large extent. This observation can be reconciled with the formula suggested if it is taken into account that the composition of the complex which is formed, as determined by Job's method, is independent of the equilibrium constant of the complex. Thus, the complex may be largely dissociated, and only with further addition of the organic anion will the dissociation be repressed sufficiently to give a considerable decrease of total cationic complexes.

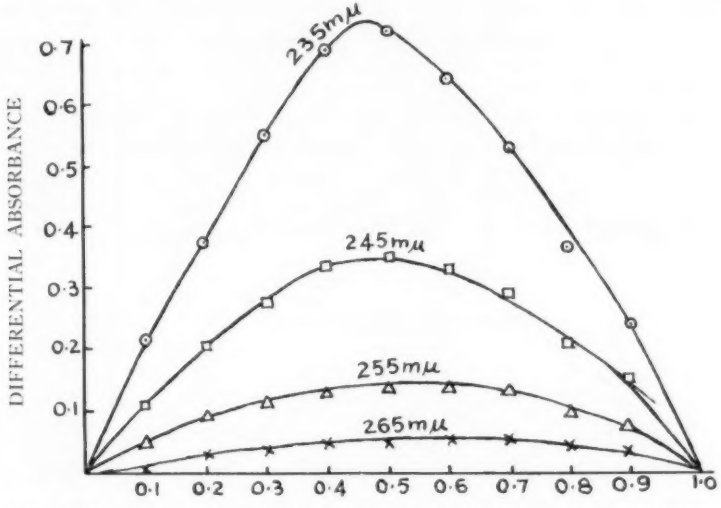
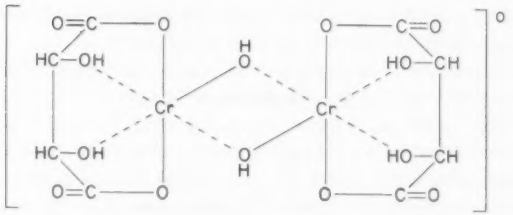


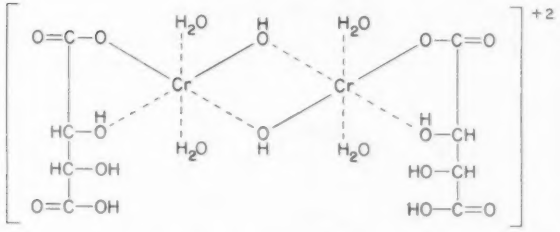
FIGURE 16.—Absorbance of chrome liquors treated with tartaric acid and sodium tartrate at pH 3.2; continuous variation method.

Fig. 16 gives data for Cr–tartaric acid–tartrate solutions adjusted to constant pH. There is a well defined maximum at the 0.47–0.48 ratio, corresponding to a complex containing 1 tartrate and 1 Cr. The curve is very symmetrical on both sides of the maximum at all wave lengths; thus the spectrophotometric readings point to the formation of a single complex of this composition at this pH with 1/3 basic chromium sulfate liquor. The tartrate complex may therefore be represented as follows:



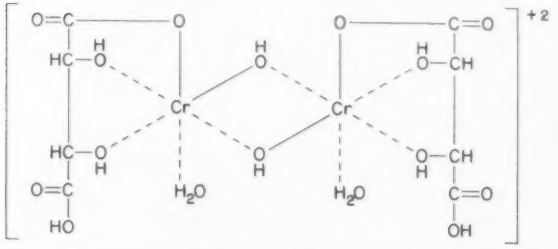
(IV)

(If both carboxyls and both hydroxyls coordinate.)



(V)

(If only one carboxyl and one hydroxyl coordinate.)



(VI)

(If one carboxyl and both hydroxyls coordinate.)

In Formula IV both the carboxyl groups are ionized and coordinated to Cr, the net charge becoming 0. Since the second pK value of tartaric acid is of the order of 5, it cannot be said with certainty that the second carboyxl group is ionized enough to permit coordination. Further, the steric factors involved in the formation of such a complex are not known.

It is pertinent to refer to the data obtained on the addition of tartaric acid alone to the chrome liquor, when an indication of a 1:2 complex was obtained. The explanation for the higher ratio of tartrate to Cr probably is that the carboxyl group which is not ionized because of the low pH does not coordinate. This permits another molecule of tartaric acid to coordinate. Deolation and partial displacement of OH groups are also probable because the displaced

sulfuric acid remains in the system whereas in the experiments at pH 3.2 this acid has been neutralized by alkali in adjusting to constant pH.

In the case of citrate and a mixture of citric acid and citrate (Fig. 17) the composition at the well defined maximum shows a complex containing 1 Cr and 1 citrate. The curve is extremely symmetrical on both sides of the maximum at all wave lengths, and the same maxima were obtained even when sodium citrate alone was added, the pH increasing to 4.6 at the maximum and to a value of 6.8 for one solution. Hence the data show that a very stable 1:1 complex is formed.

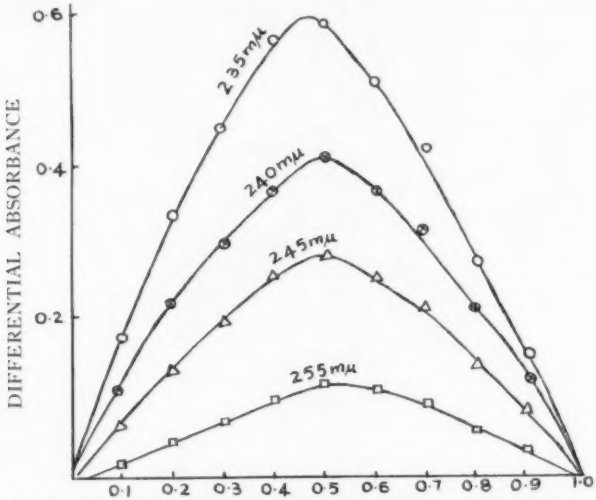
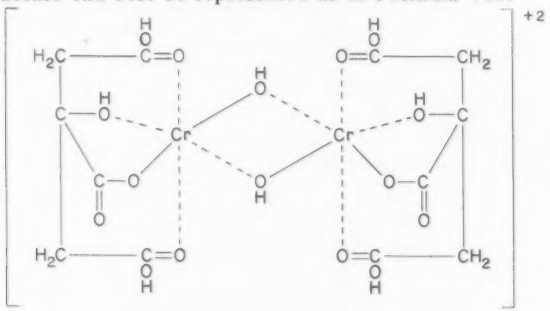


FIGURE 17.—Absorbance of chrome liquors treated with citric acid and sodium citrate at pH 3.2; continuous variation method.

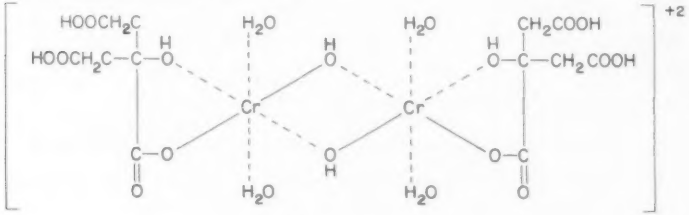
The structure can best be represented as in Formula VII:



(VII)

The steric effects on the stability of such a complex are not known. Since the formation of 6-membered rings containing one double bond involves less strain than without any double bond (21) all the 6-rings are drawn with one double bond. This method of representation implies that the ionization of the other two carboxyl groups is no criterion for the formation of this complex. In fact since the pK values of the second and third carboxyl groups of citric acid are fairly high ($pK_2=4.7$; $pK_3=6.4$), it is quite probable that they do not coordinate through their ionized COO^- groups. The charge of the complex essentially depends upon how the carboxyl groups are linked to Cr, whether through the negatively charged oxygen or through the C=O group.

The alternate method of representation of the formula, with the olated OH groups undisturbed, is to assume that the unionizable COOH groups are not coordinated, as follows:



(VIII)

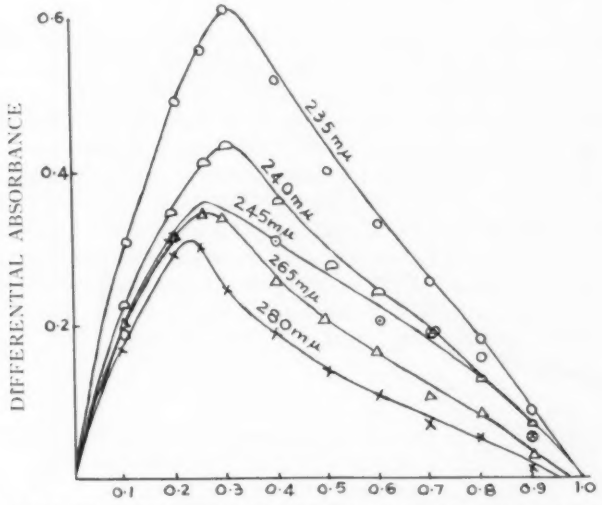
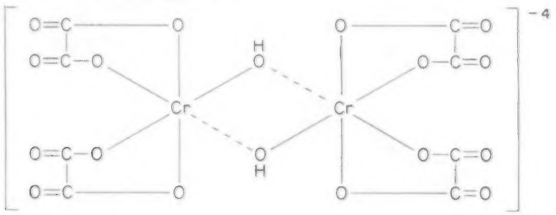


FIGURE 18.—Absorbance of chrome liquors treated with oxalic acid and sodium oxalate at pH 3.2; continuous variation method.

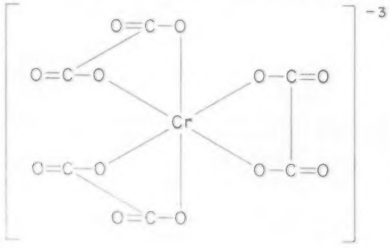
In this formula the 6-membered ring does not find a place, but the charge remains zero. The only probable objection to this structure is the question why another citric acid molecule does not coordinate displacing the aquo groups and forming a complex containing 2 citrates to 1 Cr. Whether or not another molecule can be added to the complex by coordination is decided by the comparative stability of the two complexes and the steric hindrance effects involved. Further light on these factors is necessary to predict the true structure.

Fig. 18 gives the curves for oxalic acid–oxalate liquors at different wave lengths. Two maxima are discernible, one at the 0.32 ratio, exhibited at wave length near 235–240 mμ, and the other at the 0.23–0.25 ratio at wave lengths of 245–280 mμ. The dioxalate complex of the following formula (IX), corresponding to 1 Cr and 2 oxalate, is indicated, since absorption at 235–240 mμ is specific for OH groups.



(IX)

However, the curves at higher wave lengths indicate trioxalate formation. It has also been observed that if the pH is slightly higher (3.6–3.7), only the trioxalate complex is indicated, and no maxima corresponding to the 1:2 complex are obtained from spectrophotometric readings. Since the oxalate is highly penetrating and the pH of the liquor used is slightly above that of the original chrome liquor, it is not unreasonable to expect the displacement of one OH group. In the case of a few solutions, an increase in the pH after complex formation was observed, confirming this supposition. It is significant that even when oxalic acid alone was used, there was some indication at high wave lengths of formation of the 1:3 complex. The results indicate that both tri- and dioxalate complexes are simultaneously formed. The trioxalate complex is represented in the usual way (Formula X):



(X)

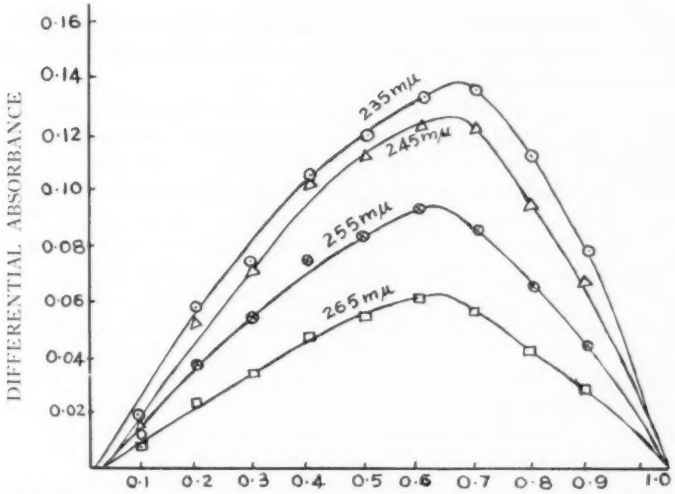


FIGURE 19.—Absorbance of chrome liquors treated with aminoacetic acid (glycine) at pH 3.2; continuous variation method.

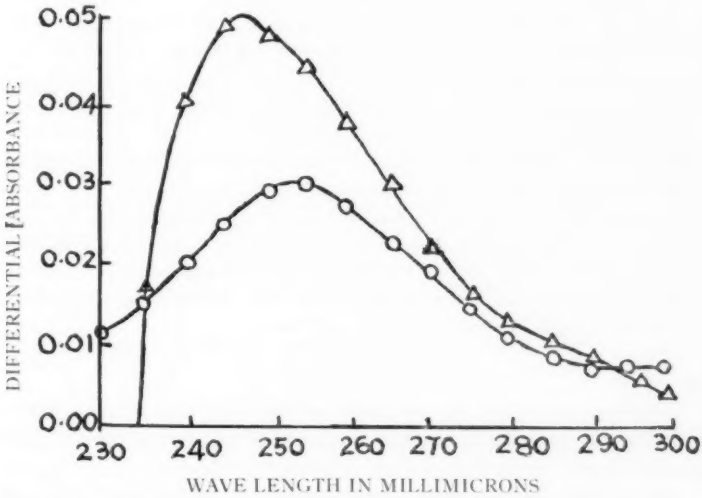


FIGURE 20.—Differential absorbance at various wave lengths between deolated chromium sulfate solution and chromium sulfate solutions treated with acetic acid.

△ pH not adjusted
○ pH adjusted to 3.2

The differential absorbance readings in the case of liquors containing oxalic acid showed a maximum at 265 mμ which was exhibited also by a pure specimen of dioxalatochromiate.

The results obtained with aminoacetic acid when adjusted to pH 3.1 are given in Fig. 19. At all wave lengths maxima occur at the 0.65–0.67 ratio corresponding to a complex containing 1 aminoacetate to 2 Cr. With aminoacetic acid only, which gives a higher pH (5.5), an indication of a 1:1 complex had been obtained. In interpreting the results to give some insight into the nature of the complex, the question arises whether amino groups coordinate with chromium or not. Shuttleworth (22) is of the opinion that the amino group does not coordinate with Cr and hence cannot form a chelate ring, whereas Küntzel (23) and Gustavson (24) believe that NH_2 is capable of coordination with Cr. Recently Green (25) has shown that phenolic and amino groups play a considerable part in the tanning of acetylated collagen.

The spectrophotometric results obtained in the ultraviolet indicate that if the pH is maintained at 3.2, aminoacetic acid is linked to the Cr in the molar ratio 1:2. This may mean that the amino group is attached to one Cr atom and the acetate to the other Cr, the two Cr atoms being already linked by OH groups. The coordination of both the amino and the carboyxl groups to the same Cr atom should give at least a 1:1 complex, which is not observed at a pH of 3.2. Alternatively, if linking takes place through only one group, e.g., the carboyxl, only random linkage is possible, and the net effect cannot show itself as a 2:1 complex. However, the difference in the data obtained between aminoacetic and acetic acids is very significant and must be attributed to the coordination of the amino group. The representation of the formula is difficult because of the low ionization of the carboxyl group at pH 3.2 and presence of positively charged NH_3^+ groups. Differential absorption spectra between the Cr solution and the boiled solution of the same concentration with acid added exhibited a maximum at 240 mμ in the case of aminoacetic acid, while for formic and acetic acids this maximum was at 250–255 mμ. Polarographic data (26) also have brought out the difference between Cr-acetic acid and Cr-aminoacetic acid complexes—the latter exhibiting a third wave. The true picture of the aminoacetic acid complex must await further evidence.

SUMMARY

1. Complex formation in chromium sulfate solutions treated with organic acids and their salts has been studied by measuring the differential absorbance in the range from 230 to 350 millimicrons.

2. The results obtained with formic, acetic, and chloroacetic acids, while providing qualitative evidence for complex formation, do not permit definite conclusions to be drawn as to the composition of the complexes that are formed.

3. Definite evidence has been obtained for the formation of complexes containing 2 glycolate, 1 tartrate, 1 citrate, and both 2 and 3 oxalate molecules per atom of chromium. Various alternative structural formulas for these complexes are presented and discussed.

4. Aminoacetic acid (glycine) appears to form a complex containing 1 glycine molecule and 2 Cr atoms, at pH 3.2; at higher pH values the ratio is 1 : 1. Possible structures for these complexes are discussed.

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The British Museum has an Egyptian collection which includes a workman's leather apron with a small purse-shaped pocket, as well as several types of leather shoes and sandals, and rolls of maroon colored leather.

—*Economics of Cattlehide Leather Tanning.* Merrill A. Watson, D.C.S. Chicago: Rumpf Publishing Co., 1950.

ENGLISH STYLE IN SCIENTIFIC PAPERS*

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Many scientific papers published in Great Britain are written in a style quite different from that adopted by good English authors. There would be no disadvantage in that, if an improved version of our language had been invented for scientific papers. This, however, is not so. For clarity and directness one must turn to nonscientific authors. One hesitates to direct attention to this subject lest one be accused of setting oneself up as a stylist. There is no such intention here. It is proposed merely to suggest a few ways in which scientific papers could be made simpler, clearer, and more pleasant to read.

To prevent any misunderstanding, it must be remarked at the outset that it is no part of my purpose to try to oppose the use of necessary scientific words. On the contrary, the precise use of certain technical terms, carefully defined and internationally understood, is an obvious necessity. We shall be concerned here with the words that stand between the technical terms.

If one examines all those passages in scientific papers that are least in accord with good English style, one finds that there are three main kinds of error, which may be described briefly as those of grammar, grandiloquence, and German construction.

It would be absurd to treat the subject of grammar at any length here, since good advice on this subject is so readily available. Grammatical errors in scientific papers sometimes make it impossible to be sure what a sentence is intended to mean. In other cases the mistakes are troublesome only because they distract the reader's attention from the subject under discussion. The words ending in *-ing* (especially *using*) are very frequently misused. One repeatedly finds such statements as this: "Examining a capillary under the microscope, after staining with carmine, it resembles a homogeneous hyaline tube." Yet anyone who is intelligent enough to carry out scientific research at a university can easily grasp in 15 minutes everything that it is essential to know about the use of present participles and gerunds.

The menace of grandiloquence seems to derive from the time when doctors used many Latin words; and indeed these errors are much more common in medical and biological than in chemical and physical writings. It is difficult to account for the fact that an Englishman will write "*vide supra*" when he means "see above." It is noticeable that those who use Latin tags most freely

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do not always know enough of that language to realize that the word data is plural.

Long words derived from Greek or Latin roots are often used for expressing very simple ideas. When a man wants to say that something is visible to the naked eye on merely opening the body-cavity, he tries to make it sound grand by saying, "this phenomenon can be macroscopically observed upon laparotomy." Sometimes he uses short words, but chooses an elaborate way of expressing himself. Thus, if he wants to refer to a sheet of metal about 1/100 in. thick, he says that it is "of thickness $\sim 10^{-2}$ in." If his intention is to tell his readers that the longest of the six pairs of chromosomes in a cell is about three times as long as the shortest, he writes, "the maximum size range of the six pairs as represented by the ratio length of chromosome 1/length of chromosome 6 is about 3/1."

The grandiloquent writer brings in abstract words where none is required. Instead of writing "because the surface of the retina is spherical," he substitutes "because of the sphericity of the retinal surface." Instead of saying that something is near the nucleus, he says that it "occupies a juxta-nuclear position."

Genteelism is allied to grandiloquence. People who think it polite to call a napkin a "serviette" have their counterparts in science, who cannot soil their mouths with paste or mash but must say "Brei" instead. Another example is "sacrificed" for "killed." Allied to genteelism is the deliberate use—not just occasionally, but over and over again—of archaic words instead of their exact equivalents in plain modern English (for example, "save" for "except").

Vogue-words have their place in the vocabulary of the grandiloquent; but what may be called negative vogue-words must also be mentioned here—perfectly good words that for an inexplicable reason must not be used. Why must the word *about* never be used by the grandiloquent? Why must they always say "*circa*," "*ca.*," "*c.*," "approximately," "around," "of the order of," and even "$\sim$"? What is the objection to "separate"? Why must "discrete" invariably be substituted? (A case can be made for the occasional use of "discrete," when special emphasis on complete separation is necessary.) *After* is another negative vogue-word.

Grandiloquence and related foibles, however, are not the worst enemies of good English. A still more serious and more insidious evil exists.

In ordinary speech writing it would never enter anyone's head to say "a tea containing cup"; one would naturally say "a cup containing tea." Yet in scientific journals one will find (for example) "iron containing globules," when what is meant is "globules containing iron." It is questionable whether the writers of such phrases have ever asked themselves why they adopt this construction, which is strange to our language and does nothing to improve it. Why do they say "a hyaluronidase treated area" when they mean "an area treated with hyaluronidase"? Why place a complex adjectival phrase before

the noun, and why omit the clarifying preposition? Why say "nonformalin containing fixatives" instead of "fixatives not containing formalin"? Why "eight micra thick sections" when one means "sections 8 μ thick"? Or why does a man who would never dream of saying "the Jones associated people" write of "the nucleolus associated chromatin"?

These, however, are relatively innocuous examples, compared with many that disfigure our scientific journals. "A methylene blue stained preparation of rabbit ear skin" is bad enough, but not quite so bad as "Dytiscus red flight muscle sections" or "the use of acidified (by HNO_3) or alkalized (by ammonia) solutions of silver." Even these, however, are not the worst English that it is possible to write, if one is determined to pile up qualifying words other than adjectives in front of the noun they qualify. The limit is perhaps reached by "adenosine triphosphate activated actomyosin contraction." (To spare the reader trouble, I may mention that this means the contraction of actomyosin, activated by adenosine triphosphate.) These are words put together without the slightest attempt at clarity or any consideration for the reader. The phrase is worse than merely illiterate: it is rude.

This piling-up before a noun of words that are not adjectives, but are used adjectivally, is rather a new fashion in English scientific writing. It was seldom seen in Britain a quarter of a century ago. There can be scarcely any doubt about its origin. That many American scientists are of German descent is obvious when one looks at lists of authors in journals published in the United States. The process of piling up adjectival phrases is familiar to everyone who knows the German langauge. It does not make for clarity. It comes naturally to a German to write "die einzigen, durch unmittelbare Beobachtung sicher zu ermittelnden Stellen" or "das von dem Objectiv-system in der der Lichtquelle zugeordneten Ebene entworfene Beugungsspectrum." Unnecessarily difficult though this construction may seem to an Englishman, yet the German language does at least help us, through the inflection of the words, to follow our path through such passages as these. But when this kind of construction is forced artifically on our much less inflected tongue, and the prepositions are omitted, all pretence of clarity is lost.

The German-American style did not originate with men who had set themselves the high ideal of improving our language as a vehicle for scientific ideas. It was, on the contrary, the product of childhood, introduced by people whose parents had spoken in sentences constructed in their native, German way. It is strange indeed that an Englishman or Scot, accustomed since infancy to a simple syntax, with logical order of words, should copy a German-American imitation of English just because he happens to be writing in a scientific journal. Our own language, as written by those who know and love it, can scarcely be surpassed for clarity, directness, and simplicity.

It is noticeable that scientific books published in the United States are often written in a style that is close to ordinary English (though the Ameri-

can people have their own special words, spellings, and phrases). The difference in style between American books on one hand and papers on the other suggests that the publishers correct the style of writing where necessary while the editors of journals do not. It must be remarked that the German influence is altogether lacking from some American scientific papers which are models of lucidity and good style. A paper by Michaelis (1) on the reaction of dyes with nucleic acids may be quoted as a particularly fine example.

How can the standard of English in scientific journals be improved? One thinks at once of the obvious works of reference—of Fowler (2), Partridge (3), and Gowers (4). Quiller-Couch's lecture "On jargon" (5) is packed with good advice and horrid examples. The teachers of English in our schools could help very much, if they were to study the errors made in scientific papers and base their instruction to science students on their findings. Style, however, is largely dependent on example. Good reading makes good writing. One could almost imagine that some of the contributors to our scientific journals had never read anything but German-American. Yet good examples abound, in books of widely different scope. A trio so diverse as Macaulay, P. G. Wodehouse, and Sir Winston Churchill have this in common, that their English is lucid. There is no dearth of good examples to suit every taste.

A strange fact that gives some basis for optimism is this. When an author has finished the scientific part of his paper, he often addresses a note of general information to the reader. At this point he suddenly discloses for the first time that he can write English, for his ideas are clearly expressed. If one wished to translate what he now says into the style adopted in the rest of the paper, one would have to write something like this: "Some related interest possessing observations by the present writer *et al.* will be the subject of *ca.* $10^{0.3010}$ discrete communications" (6). The fact that he does not write like that shows that he need not have done so in the body of the paper.

One last necessary word. The best English writers occasionally use some of the strange constructions mentioned in this article, often to produce a special effect for a particular occasion. They do so, however, only at long intervals. The greater part of their writing is so smooth and fluent that the reader forgets that he is reading and knows only that he is absorbing ideas.

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6. With the exception of the passage in quotation marks in this paragraph, all the examples given in this article are genuine: none was invented by myself or modified to suit my purpose. I am indebted to A. C. Hardy for a careful criticism of the first draft of this article.

LIFE LINES

Brief Biographies of Our Contributors

S. R. EVELYN was appointed a student assistant at the Leather Industries Research Institute, Grahamstown, South Africa, while studying for a B.Sc. degree at Rhodes University College. After graduating in 1949, he was awarded a scholarship by the Leather Institute for two years at Birmingham University, England. He obtained the M.Sc. degree there in 1953 and was awarded the 1955 Donald Burton prize by the S.L.T.C. for his papers on the molecular weight of wattle tannins.

DR. Y. NAYUDAMMA, an active member of this Association, is known personally by many of our members because of his residence at Lehigh University in the late 1940's. His undergraduate training was received at Benares University. He was a research chemist at the Institute of Leather Technology, Washermanpet, India (1943–45), after which he studied at the College of Technology, Northampton, England. From 1946 to 1951 he studied under the late Prof. E. R. Theis at Lehigh University, where he received his doctorate. Since returning to India in 1952 he has been associated with the Central Leather Research Institute, Madras, of which he is Assistant Director. He is the author, or coauthor, of many papers. He is a member of numerous scientific and technical societies, including the A.L.C.A., the S.L.T.C., and the American Chemical Society; an Associate of the Royal Institute of Chemistry (England) and a Fellow of the Institute of Chemists (India); and a Fellow of the Sigma Xi Society.

DR. D. RAMASWAMY received his B.Sc. degree from St. John's College, Jaffna, Ceylon in 1946. In 1947 he joined the staff of the Indian Institute of Science, Bangalore, where he worked for six years on various projects in inorganic and physical chemistry. He was awarded the M.Sc. and Ph.D. degrees by Madras University on the basis of theses submitted. He joined the Central Leather Research Institute, Madras, in 1953, and is working on the application of instrumental methods to problems in leather research. These studies have resulted in the publication of several papers, such as the one in this number.

DR. D. G. ROUX graduated from the University of South Africa in 1945 with an M.Sc. degree (distinction) for his work on essential oils of rutaceous plants. After a brief period as research chemist to the United Tobacco Company, Cape Town, he joined the staff of the Leather Industries Research Institute, Grahamstown, in 1947, where at present he holds the post of Senior Research

Officer. Dr. Roux has specialized in the chemistry of wattle tannins and has published works on tannins, catechins, and anthocyanidins in British, German, and American chemical and leather literature. He received the Donald Burton Prize (1950), the Ph.D. degree of Rhodes University (1952), and the Fellowship of the Royal Institute of Chemistry (1955). During 1954, on a travel grant from the Leather Institute, he studied catechin chemistry with Professor Karl Freudenberg at the Chemical Institute, Heidelberg University, Germany, and chromatography at the Low Temperature Research Station, Cambridge, England.

In a Berlin museum . . . there is a stone carving, reputed to be more than 4000 years old, showing Egyptian leather dressers at work.

—*Economics of Cattlehide Leather Tanning.* Merrill A. Watson, D.C.S. Chicago: The Rumpf Publishing Company, 1950.

The Metropolitan Museum of Art in New York City has on display a remarkably well preserved pair of Egyptian leather sandals more than 33 centuries old.

Ibid.

At Cairo is preserved the funeral tent of an Egyptian queen of the twenty-first dynasty (circa 1100 B.C.). Several pieces of gazelle hide of various colors had been stitched together to make this tent, producing a surprising mosaic of leather work.

Ibid.

Pliny recorded the fact that hides were tanned with bark and that gall nuts, sumac, and lotus were used (in tanning).

Ibid.

With few modifications, the tanning processes of the late Roman era introduced the basic principles of vegetable tanning which are followed even by the modern tanners.

Ibid.

[In medieval Europe] One of the earliest guilds of record was that of the leather workers of Florence, first mentioned in the archives of the States in the year 1098. By 1238 the industry had become well organized and was placed 11th in order of importance among guild organizations. In 1282 the name of the guild was changed to the Guild of the Master Tanners. By that time the tanners' guilds had developed a high stage of organization with a chief officer and four agents who fixed the prices of hides, skins, and leather, and the rates for tanning and dressing, and who kept records of all workmen employed by the guild.

Ibid.

ALCA NEWS

Proceedings, Reports, Notices, and News

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EMPLOYMENT NOTICE

WANTED—position by member. Tanning control chemist desires to relocate in Los Angeles, California, area. Wide experience in tannery process and production control. Tannery liquors, finish binders, pigment grinding and control, tannery air conditioning, paste and pasting unit control, water control and conservation, laboratory methods and procedures, process engineering. Permanent position in progressive tannery desired. Please direct reply to Box TW184, c/o Secretary, American Leather Chemists Association, University of Cincinnati, Cincinnati 21, Ohio.

REPORT OF THE HIDE POWDER COMMITTEE

The sample of the hide powder furnished by Mr. Frank F. Marshall, Ridgway, Pennsylvania, and representing a batch designated as Lot No. 28, has been tested by the A.L.C.A. Hide Powder Committee. Average values found by the Committee members in certain comparative tests with A.L.C.A. Standard Hide Powder Lot No. 27 and the proposed Lot No. 28 were as follows:

Hide Powder Lot	No. 27	No. 28
Date of testing	September, 1956	September, 1956
Water-soluble content,		
Blank test residue (grams)	0.0030	0.0031
Ash, %	0.21	0.21
pH	5.44	5.38
Nontannin, grand average, %	19.99	19.85

The Committee has approved the new hide powder. Mr. Frank Marshall was authorized December 10, 1956 to issue the hide powder represented by the sample as A.L.C.A. Official American Standard Hide Powder Lot No. 28.

G. V. Downing	F. F. Marshall*
G. E. Daub	G. W. Schultz
A. N. Kay	I. D. Clarke, *Chairman*

*Participated in the testing but not in the approval of the hide powder as official.

SECRETARY'S NOTICE

The library of the late Editor Dean Williams is for sale, collectively or in part. It comprises:

Chemical Abstracts	1910 – 1955
Journal of the American Chemical Society	1914 – 1955
Industrial & Engineering Chemistry - Industrial Edition	1912 – 1955
Industrial & Engineering Chemistry - Analytical Edition	Vol. I – 1953

Some numbers are missing.
Address inquiries or offer to

Mrs. R. Dean Williams
219 Park Avenue
Ridgway, Pennsylvania

CANDIDATES FOR THE 1957 ELECTION

CANDIDATES FOR COUNCIL

David Balfour
Charles A. Martin
Harold R. Miller, Jr.
Herbert A. Tetreault
Harry J. Widney
C. David Wilson

The above slate of candidates has been prepared by the Nominating Committee.

ARTHUR N. KAY, *Chairman*
Reuben Henrich
Dominic Meo

Members please note the following constitutional provision:

Article V-2, Nominations (c).
Additional candidates may be nominated by petitions, signed by 15 or more active members. Such petitions shall reach the Secretary not later than April 1, and shall contain a statement of the willingness of the nominee to serve if elected. No member shall sign more than one petition for each office to be filled.

An indigenous, crude art of making sole leather was developed gradually in Western Europe from the use of raw hide for shields. The Flemish, especially, developed considerable skill in the making up of such leather, but the leather itself was made mainly as an adjunct to the farming operations by leaving hides for many months in an ooze of oak bark. In fact, three years was not an unusual period for the hides to be left in the tan.

—*Lactic Acid in Leather Manufacturer*, Alan A. Claflin and Harry P. Corson. Boston: The Avery Chemical Company, 1920

In the United States approximately 500 plants, ranging from modern industrial enterprises to little more than handicraft establishments, process various kinds of hide and skins into a great variety of leathers. Despite the advance in technology and the growing use of synthetics, the production of leather remains an important industry.

—*Economics of Cattlehide Leather Tanning.* Merrill A. Watson, D.C.S. Chicago: Rumpf Publishing Co., 1950.

ABSTRACTS

Sodium Gluconate as a Complexing Agent in the Volumetric Analysis of Aluminum Compounds. H. Watts and D. Utley. *Anal. Chem.*, **28,** 1731 (1956).—Sodium gluconate satisfactorily replaces both tartrate and barium in the titration of aluminum and has these advantages: (1) use of a single complexing reagent, (2) lack of sulfate or carbonate interferences, and (3) the titration of carbonate to the bicarbonate, in the presence of aluminum, in a buffered solution. The effects of impurities are pointed out, and tables showing the accuracy of the proposed method are given. H.R.W.

Identification of Amines as Tetraphenylborates. F. Crane, Jr. *Anal. Chem.*, **28,** 1794 (1956).—Basic organic nitrogen compounds are detected by precipitation of the tetraphenylborate compounds in aqueous acid solutions. The amine is identified by determining the melting point of the washed, dried tetraphenylmetaborate precipitate. Quantitative determination of the amine is done by (1) the use of a new potentiometric method, based on the solubility of silver tetraphenylmetaborate in aqueous acetone, to obtain the TPB content of the compound, or (2) the analysis of the borate derivative for nitrogen by the conventional Dumas method. H.R.W.

Mechanism of Gelation of Gelatin. Influence of Certain Electrolytes on the Melting Points of Gels of Gelatin and Chemically Modified Gelatins. J. Bello, H. Riese, and J. Vinograd. *J. Phys. Chem.*, **60,** 1299 (1956).—The effects of a long series of salts and acids upon the melting point of 5% gelatin gels show a correlation between the binding of ions, as indicated by pH changes, and the resulting melting points. With the use of chemically modified gelatins it is shown that the binding of cations or anions at specific terminal groupings is not the cause of the melting point reductions. The results of this work support other reported findings that polarizable anions reduce the melting points in varying amounts. H.R.W.

Convert Michigan Sawmill Wastes into Leather Tannin. USDA Agricultural Research Service. *Leather Mfr.*, **73,** No. 10, 34 (1956).—Hemlock slabs are largely wasted because of the difficulty of separating the bark from the wood. When the slabs are seasoned from 7 to 10 months (to reduce H_2O to <20%) a certain type chipper very efficiently detaches the wood from the bark. An air-flotation specific gravity separator separates the slabs into an 80% bark product containing 10% tannin and a 95% wood chip product. Tannin extracted from the bark chips and concentrated is similar in composition to the traditional peeled hemlock bark tannin. The wood chips slightly contaminated with bark can be made into fine bleached paper equal to any obtainable from regular peeled hemlock pulpwood. A detailed description of the process is published in a recent issue of *Forest Products Journal*, Forest Products Research Society, Madison, Wisconsin. J.J.

Coated Abrasives—Their Use in the Tanning Industry. C. L. Flooding. *Shoe and Leather Reporter*, **284,** No. 1, 10 (1956).—There are three

basic components in a coated abrasive—the mineral, the bond, and the backing. The minerals used in coated abrasives for tanneries are flint, garnet, aluminum oxide, and silicon carbide. The bond is resin over glue on fiber-backed and light-weight sandpaper; this bond will not soften under high frictional heat, and the tendency toward filling is greatly reduced. Animal glue is also used as a binding agent, but it will soften under high frictional heat. The backing commonly used is "E" weight paper (approximately 130 lb. per ream) or vulcanized fiber about 20 mils thick. The paper is more flexible whereas the fiber is stronger. Flint paper is generally used in fine grit paper (280, 320, and 400), garnet in grits of 80 to 220, aluminum oxide in grits of 180 to 400, and silicon carbide in grits of 240 to 400. J.J.

Hide Industry Takes Big Step Forward. Anon. *Leather and Shoes*, **132**, No. 17, 12 (1956).—A process of defleshing hides before shipment is described. Manure and foreign matter are removed by air and water under high pressure. Fleshing and tannery trimming remove about 13 pounds from a green hide. Bleach added to brine eliminates bacterial spoilage of hides for an indefinite period. Penetration of salt in brining is said to be complete in 16 hr. or less, but currently the hides are brined 48 hr. Hides from the process are odorless. Sores, grub holes, brands, deep scratches, etc., are visible after fleshing. The hides shrink about 12% in curing. Freight saving on a carload of 1,000 hides is $135 to $195. J.J.

Studies on the Shrinkage Phenomenon. II.—Tanning with Formaldehyde. Y. Nayudamma and T. S. Krishnan. *Bull. Central Leather Research Inst., Madras*, **2**, 397–401 (1956); cf. *JALCA*, **51**, 678 (1956).—Shrinkage temperatures were measured on leathers tanned with HCHO and retanned with various agents, and on leathers tanned with these same agents and retanned with HCHO, with the following results:

Tanning Material	Shrinkage Temperature, °C.		
	Single Tannage	Retanned with HCHO	Pretanned with HCHO
Formaldehyde	83	.....	.....
Basic Cr sulfate	120	122*	112
		125†	
Basic Al sulfate	85	88	84
Wattle extract	90	107	88
Myrobalans extract	81	93	83.5
"Basyntan"	65.5	79	64
"Immergan" (RSO_2Cl)	58	83	74.5
Fish oil	64.5	86.5	76

*Retanned at pH 7 †Retanned at pH 4

Retannage of the mineral-tanned leathers with HCHO increases T_s very slightly, while pretannage with HCHO results in a slightly lower T_s than is obtained with Cr or Al tannage alone. This may be due to inactivation of NH_2 groups which act as acid acceptors in mineral tannage. The striking increase in T_s for vegetable-tanned leathers retanned with HCHO may be due to cross-link-

ing by HCHO of tannin molecules fixed by collagen. Pretannage with HCHO seems to have little effect on T_s of vegetable-retanned leather. The low T_s values for leathers tanned with Basyntan, Immergan, and fish oil indicate that these tannages produce little if any cross-linking. Retannage with HCHO raises the T_s to values approximating those obtained by tanning with HCHO only, indicating that HCHO is fixed by groups other than those which combine with the 3 non-cross-linking tanning agents. Leather pretanned with HCHO and retanned with Basyntan has about the same T_s as Basyntan leather, indicating displacement of HCHO by the acid syntan. A similar but less marked effect occurs when HCHO-pretanned leather is retanned with Immergan or fish oil. H.B.M.

Combined Use of Masking Agents in Chrome Tanning. Y. Inouye, Y. Oono, and Y. Fukui. *Bull. Japan Assoc. Leather Technol.*, **2,** 81–86 (1956) (English summary).—The effects of adding Na formate, Na_2SO_3, and Na phthalate, singly and in pairs, on fixation of Cr and T_s of leather were studied. A pH value from 4 to 5 is necessary to get high Cr fixation and T_s with formate liquors. At these pH values, Cr fixation in 48 hr. declines slightly with increasing amount of formate but increases with increasing sulfite and phthalate. T_s does not always increase with increasing Cr fixation. T_s increases with the amount of formate and sulfite added, but declines with amounts of added phthalate. Phthalate and sulfite used together are more effective than either alone. The effect of formate plus sulfite is about the same as phthalate plus sulfite when the ratio of formate to sulfite is small. Formate plus phthalate is about as effective as phthalate alone. Aging lowers the tanning potency of phthalate-masked liquors. Formate masking produces fullness and softness; phthalate produces fullness; small additions of sulfite produce a smooth grain, but larger additions produce drawn grain. Phthalate liquors are stable at high pH values, but at too high pH values the leather shows drawn grain. (*Note:* The tables in this article have English headings and are easy to understand). H.B.M.

Properties and Applications of Sequestering Agents. R. L. Smith. *Chem. and Industry* **(1956),** 1284–91.—An address dealing with the chemistry of chelating agents and their applications in industry. H.B.M.

Some Considerations on Basicity of Chrome Liquor as the Standard of Chrome Tanning. A. Kawamura and K. Wada. *Bull. Japan Assoc. Leather Technol.*, **2,** 71–80 (1956) (English summary).—The authors believe that the role of basicity in chrome tanning has been over emphasized. The amount of alkali ordinarily added in neutralizing has little effect on the charges and complexly bound SO_4 content of Cr complexes. Merely heating $Cr_2(SO_4)_3 \cdot 6H_2O$ solutions for 30 min. brings them to the same colloidal state as found in 33% basic, SO_2-reduced Cr liquor. The structure of Cr complexes ("true" colloidal state, charge distribution, etc.) seems to be an important controlling factor in tanning. H.B.M.

Chamoising Experiments with Pelt of Superannuated Sheep. Y. Sakimoto. *Bull. Japan Assoc. Leather Technol.*, **2,** 87–91 (1956) (English summary).—Fairly good leather was produced from skins of old sheep. Four

different fish oils were used. The tanning capacity of the oil depended on its content of unsaturated, oxidizable fatty acids as well as its I_2 value. No relationship was found between leather quality and so-called combined-oil content. The rate of replacement of water by oil in the skin had more effect on the rate of chamoising than chemical pretreatments of the pelt with HCHO, acids, bichromate, and chlorate. H.B.M.

Rapid Procedure for Estimation of Amino Acids by Direct Photometry on Filter Paper Chromatograms. L. Rockland and J. Underwood. *Anal. Chem.*, **28,** 1679 (1956).—An improved micro-method which consists in employing a special device for mounting the small-scale filter paper chromatograms in front of the photocell of the photometer. H.R.W.

Determination of the Hydrophile-lipophile Character of Surface-active Agents and Oils by a Water Titration. H. Greenwald, G. Brown, and M. Fineman. *Anal Chem.*, **28,** 1693 (1956).—The requirements of emulsifiers are discussed briefly. Titration with water of a dioxane-benzene solution of oils or surface-active agents to a cloud end point gives a useful index of their suitability as emulsifiers. A list of the best single or mixed emulsifiers for oil-water systems is given. H.R.W.

The Formation of C^{14} Hydroxyproline from C^{14} Proline by Collagen-forming Cells *in Vitro*. R. H. Smith and S. F. Jackson. *Biochem. J.*, **64,** 8P (1956).—Experiments were devised to study the ability of collagen-forming tissue to hydroxylate L-proline *in vitro*. The amount of hydroxyproline formed during 24-hr. exposure to C^{14} L-proline varied with the age of the culture and the corresponding development of extracellular material; 24-hr. cultures gave 1% or less conversion and 5-day cultures approximately 5%. J.M.C.

Studies of the Tanning Characteristics of Modified Waste Sulfite Liquors. K. B. Mathur, S. K. Barat, and B. M. Das. *Bull Central Leather Research Inst., Madras,* **3,** 23–34 (1956).—This paper describes experiments with condensation products of Na lignosulfonate with resorcinol (cf. *JALCA*, **46,** 301 [1951]; **50,** 85 [1955]). Na lignosulfonate was prepared from a paper mill liquor containing Mg lignosulfonate by precipitating Mg with Na_2CO_3, settling, centrifuging, adjusting to pH 7, and spray-drying. Equal weights of the lignosulfonate and resorcinol were mixed with a little ethyl ether to form a paste, treated with 10% conc. HCl on the lignosulfonate weight, and heated under reflux for 3 to 4 hr. The temperatures employed were 100°, 110°, 120°, 130°, 135°, and 140°C. The resulting resinous masses were soluble in water (only partly soluble if the temperature exceeded 140°). Degree of condensation (g. resorcinol combined with 100 g. lignosulfonate) was determined by extracting the uncombined resorcinol with ether. Degree of condensation rose from 16 at 100° to 31.5 at 120°; further increase in temperature had little effect on amount of resorcinol condensed, though there was evidence of increasing polymerization with increasing temperature. Rate and degree of tannage were measured by treating 2 g. hide powder with 200 ml. of a 20° Bk. solution of each condensate, at pH 3, for 2 and 24 hr. The tanned

powders were dried, and then soaked in 100 parts water for 24 hr., filtered, redried, and analyzed. The degree of tannage varied from 44 to 53% and was highest for the 130°C. condensate. This condensate also gave the best results in tanning tests with goatskin pieces. Rates of penetration of the different condensates into gelatin gels gave erratic results; the 130° condensate penetrated faster at pH 5 and 6 than at lower pH values. The degree of tannage from the 130° condensate determined as above declined from 44 at pH 2.5 to 28 at pH 5, and increased to 33 at pH 6. It increased with concentration, but not much for liquors stronger than 4%. The recommended tanning procedure is to tan first at pH 6 with a 15° Bk. liquor to get quick penetration; then strengthen the liquor to 30° Bk. and reduce the pH to 2.8–3.0. The color of the leather is dark, but can be improved by usual bleaching processes. The chief advantage claimed for the condensation product of resorcinol and Na lignosulfonate, in comparison with the Mg compound, is that the product is soluble without further introduction of sulfonic acid groups. H.B.M.

Physical and Chemical Characteristics of Some Indian Leathers. III.—Indian Chamois Leathers. Y. Nayudamma, T. S. Ranganathan, and B. M. Das. *Bull. Central Leather Research Inst., Madras*, **3**, 1–6 (1956).—Indian chamois leather is made mostly from spready, reject goatskins, tanned with HCHO and cod (occasionally sardine) oil. Analyses are reported for 18 samples. Results varied widely, and included some leathers that were inadequately washed (very high fat), improperly neutralized (high pH value), or loaded with mineral matter. Excluding these, the normal ranges were: H_2O, 15–20; hide substance, 65–75; H_2O-solubles, < 4; free fat, 5–10; bound fat, 1–5; insoluble ash, 2–5; total ash, 2–6; HCHO, 0–1%; pH of water extract, 6–8. Acid-soluble hide substance (*JALCA*, **45**, 810 [1950]) varied from 0.15 to 2.4%. A high grease content results in poor H_2O absorption; this may result also from incomplete removal of the grain. H.B.M.

New Barks for E. I. Tannage. II.—*Pithecolobium Dulce*. M. K. Udaya Varma, E. C. Mathew, and B. M. Das. *Bull. Central Leather Research Inst., Madras*, **3**, 7–12 (1956).—This is an abundant, shrub-like tree. A bark sample contained tannin 25%, soluble nontannin 13%, pH of analytical solution 4.8. The potentiometric titration curve is similar to that of wattle, but *P. dulce* is somewhat better buffered. Good E. I. kip leather was produced with *P. dulce* alone, but better color and firmness were obtained with a 6.5:1 blend of *P. dulce* and myrobalans. Replacement of wattle by *P. dulce* in a common 1:1 blend with konnan bark also gave satisfactory results. H.B.M.

Investigations on Making Good Leather from Dead Hides for Police and Military Boot Uppers. A. K. Sen Gupta, and B. M. Das. *Bull. Central Leather Research Inst., Madras*, **3**, 13–22 (1956).—About 85% of the available cattle hides in India come from animals that die a natural death. Because of delays in flaying and curing, these often suffer extensive bacterial damage, resulting in veiny, loose, and pipey leather. A process designed to get the best possible leather from this inferior raw stock is described in detail. This includes: a short soak or none; a short lime with Na_2S only, followed by deplumping with $CaCl_2$; conventional bate; pickling with H_2SO_4 and

$Al_2(SO_4)_3$; tanning with 29% basic Cr liquor to which Gum Tragol is added to obtain filling, followed by neutralizing as usual; retanning after shaving using a 1-bath Cr retan liquor and then neutralizing with a mixture of $Na_2S_2O_3$ and $Na_2Cr_2O_7$; coloring by conventional procedures; fatliquoring with a mixture of castor and fish oils, plus proprietary fat liquors, with the addition of casein dissolved in borax and ammonia; finishing by conventional procedures. H.B.M.

Investigations on Suint. A. J. Farnworth. *Australian J. Applied Sci.*, **7,** 233–41 (1956).—Suint is a water-soluble material present in wool in variable amount (2–9%). It contains about 1.3% Na and 26.1% K, combined mostly with water-soluble organic acids. Fatty acids are present in minor amounts only. H.B.M.

Kahua Bark in Sole Leather Manufacture. P. V. R. Krishna Reddi, K. A. Joseph, and K. Padmanabhan. *Tanner* (India), **11,** No. 4, 21–22 (1956).—Kahua (*Terminalia arjuna*) is a large tree, common throughout India. The bark can be cut repeatedly, yielding 20–100 lb. per tree every 3 years. Two samples of bark contained 20–21% tannin, 9–12% soluble nontannin, pH (analytical solution) 5.0. Both condensed and hydrolyzable tannins are present. Used alone, kahua gives a reddish, cracky leather, but a 1:1 blend of kahua and myrobalans produced satisfactory sole leather. H.B.M.

A New Substitute for Imported Wattle Bark. P. V. R. Krishna Reddi, J. A. Joseph, and K. Padmanabhan. *Tanner* (India), **11,** No. 4, 23–27 (1956).—*Cassia siamea* is an evergreen, used for ornamental purposes, easily grown, and fast growing. The bark contains about 23% tannin (condensed type), 10% soluble nontannin, pH (analytical solution) 5.4. The tannin is easily leached. Tests indicate that this tannin yields satisfactory E. I. kip leather. H.B.M.

Colorimetric Methods for the Quantitative Estimation of Formaldehyde Fixed in Collagen. S. M. Bose, K. T. Joseph, and B. M. Das. *Tanner* (India), **11,** No. 3, 23; No. 4, 16–20 (1956).—The authors treated 1 g. samples of hide powder with various amounts of HCHO at pH about 7.5 for 24 hr., then digested with H_2SO_4 and determined HCHO in the whole digest by different methods. Percentage recoveries by different methods were: Highberger-Retzsch, 97.1; Romijn-Bowes-Pleas, 96.0; bisulfite-I_2, 98.7; dimedon, 99.8; hydroxylamine HCl, 94.1; phenylhydrazine, 94.0. Colorimetric determination with Schiff's reagent in the distillate showed 97.5% recovery, but this method is applicable only if the final test solution contains more than 0.05 mg. HCHO per ml., as the color absorption curve is not linear for lower concentrations. The chromotropic acid colorimetric method (see *JALCA*, **51,** 394 [1956]) was applied directly to the digest without distillation. Recoveries varied from 98.0 to 99.9%. The method is applicable to solutions containing not over 0.09 mg. HCHO per ml. In another test, the HCHO-tanned collagen was washed, and HCHO was determined separately in the tanned hide powder, wash water, and residual HCHO tanning solution. Total recoveries were 97.4–98.3%. *Procedure:* Boil the HCHO-tanned collagen with 12 *N* H_2SO_4 for 1 hr. in an all-glass reflux apparatus, using ice water to cool the condenser. Wash

down the condenser, make up to a known volume, and dilute so that 1 ml. of the final test solution will contain not more than 0.09 mg. HCHO. To 1 ml. of the diluted solution add 0.5 ml. of a 10% solution of chromotropic acid (freshly prepared), and 5 ml. conc. H_2SO_4. Heat on a boiling water bath for 30 min., cool, dilute to 50 ml., cool again, and read the color with a photoelectric colorimeter using a No. 54 green filter. Determine HCHO by comparison with a standard curve in the usual manner. This method avoids distillation of HCHO from the hydrolyzate and is quick, convenient, and accurate. Hydrolytic decomposition products of collagen do not interfere. (*Note:* This procedure has not been shown to be applicable to leathers containing anything besides collagen and HCHO). H.B.M.

A New Look at Research. F. O'Flaherty. *Shoe and Leather Reporter*, **284**, No. 2, 31 (1956).—During the past few decades research was designed with only one variable in an experiment, but today modern research using sound statistical methods often has three or four variables in a single experiment. Modern research tools and techniques are beginning to reveal the sequence of the protein building stones (amino acids) in skin collagen and the use and effects of sound waves, gamma rays, and X rays. J.J.

Leather Research at NBS. A Summary Technical Report. Anon. *Shoe and Leather Reporter*, **284**, No. 2, 32 (1956).—The past 40 years of research activity have included studies of collagen, development of concepts for the mechanism of tanning, and measurements of the physical and chemical properties of collagen and leather. Much of the information thus obtained has been applied to the improvement of leather products and the development of standard methods for their evaluation. The report enumerates the many studies made, their importance, and how the particular studies have led to other studies on collagen and leather. Present and future work includes nondestructive sound wave testing of leather and the study of the degradation of leather by radiation. Good correlation has been found between sonic measurements and the results of tensile and breaking elongation tests. Study of the degradation by radiation will, it is believed, show the exact point of attack in the collagen molecule and thus give a clearer explanation of the mechanism of breakdown. The newly developed technique for amino acid determination will be used in this study. J.J.

The Molecular Weight of Black Wattle Tannins. III.— A Note on the Variations within and between Trees. S. R. Evelyn. *J. Soc. Leather Trades' Chemists*, **40**, 335–38 (1956).—The molecular weight variation in tannins from bark extracts of different black wattle trees was investigated. The influence of sampling at various selected heights on the tree was also determined. After quantitative extraction of the waxes, tannins were removed by extended methanol treatment. Molecular weights of tannins were determined by an ebulliometric procedure (*JALCA*, **49**, 641, [1954]). Tree-to-tree variation at breast height and at other corresponding heights was found. There was no evidence of a correlation between the height of sampling position and the molecular weight, high molecular weight tannins being present even in the youngest strippable bark. J.M.C.

The Measurement of the Wet-Firmness of Sole Leather. H. Anderson and P. J. Dwyer. *J. Soc. Leather Trades' Chemists,* **40,** 330–34 (1956).—The paper illustrates the use of a pendulum method developed by Dwyer (*JALCA,* **47,** 668, [1952]) for measuring the wet-firmness of sole leather. The leather sample is employed as the flexible fulcrum in an otherwise rigid pendulum. Use is made of a value termed "swing reduction time" which was fixed arbitrarily as the time required for the arc of swing to fall from 30 to 10 degrees. Where readings could be taken on the same sample of leather before and after treatment, a reasonably high degree of precision was attainable, and the technique differentiated the expected influences of pH, acid, and salt content. The effect of hot-pitting on the firmness of sole leather was examined. Hot-pitting exaggerated existing differences in firmness over the area of a bend of leather, but different tanning mixtures varied in the extent to which they contributed to this effect. J.M.C.

The Structure and Deformation of Collagen Fibers. II—The Morphology of Collagen Fibers. A. J. Cruise. *J. Soc. Leather Trades' Chemists,* **40,** 321–29 (1956).—The structure of collagen fibers was examined at the limits of resolution of the optical microscope by means of phase contrast, polarized light, and interferometric methods. The interferometric system proved useful in studying the hydration and dehydration of collagen bundles and in observing the reactions of individual fibers with acids and alkalies. Phase contrast was employed to study the results of crushing fibers when the material was so reduced in thickness as to be virtually invisible in ordinary light. Other noncollagenous fibrous structures were observed to be intimately associated with collagen fibers in raw tissue and in tanned material. Some of these morphological variants do not swell in acid or alkaline media, do not exhibit hydrothermal shrinkage, and resist many of the usual histological stains for collagen. This resistance to swelling, etc., is accompanied by an increase in birefringence and resistance to pressure, and the exhibition of complex structures on fracture. The proportion of these variant fibers is apparently related to the age of the specimen. J.M.C.

Study on Liming Conditions. Influence of Temperature. R. Lasserre. *Bull. Assoc. Franc. Chimistes Inds. Cuir,* **18,** 111, 147 (1956).—A study is made on the effect of temperature in unhairing of calfskin pieces in $Ca(OH)_2$ only, $Ca(OH)_2$-Na_2S, $Ca(OH)_2$-NaHS, Na_2S only, $Ca(OH)_2$-As_2S_3, and $Ca(OH)_2$-dimethylamine. The temperatures employed were 8°, 20°, 30°, and 37°C. In plain lime, pH, alkalinity, and lime solubility decrease with increasing temperature. Hair loosening begins on the third day regardless of temperature, but complete unhairing requires 10 days at 8° and only 3 to 4 days at 30°; there is no further gain in time at 37°C. Swelling decreases with increase in temperature. With increasing temperature an increasingly white pelt is obtained. Decomposition of collagen is low at 8° and 20°, more pronounced at 30°, and very marked at 37°. These results, which coincide with those of other authors, indicate that it is necessary to stay below 30°, or even 25°, to avoid excessive loss of collagen. However, the gain in time by raising the temperature from 8° to 30° is considerable, while hide substance losses remain low. The variations of the physical aspect of the pelt noticed at different temperatures are of practical interest for obtaining leather of more or less supple character. Addition of Na_2S raises the pH and decreases the solubility of lime.

Increase in temperature in lime-Na_2S has the same effect as in lime only. At the lowest Na_2S concentration (0.25%), hair slippage starts in 24 hr., and unhairing is complete in 9–10 days; with 1% Na_2S, it is complete in 2 days. Unhairing is accelerated both by raising the temperature to 30° and by increasing the Na_2S concentration; at higher temperatures the gain in time is less pronounced, and the pelt undergoes a brutal change. In all cases, increased sulfide concentration results in decreased fineness of grain but a definite increase in thickness. Addition of NaHS to lime results in increased lime solubility, with very little change in pH. Lime solubility and pH decrease slightly with increasing temperature; this effect decreases with increasing sulfhydrate concentration. Time required for unhairing decreases with increasing temperature; this effect becomes practically nil at the higher sulfhydrate concentrations, but the aspect of the pelt and hair is quite different at 8° and 37°. Since the alkalinity of the liquor is little affected by sulfhydrate concentration, no swelling occurs comparable to that obtained with Na_2S, and there is little variation in pelt thickness. A concentration of 0.75% NaHS or even higher should be considered. At such a concentration temperature has little effect on rate of unhairing and can be adjusted to obtain the character of leather desired. With Na_2S only (2 to 3%) the alkalinity decreases rapidly, and it is impossible to obtain complete unhairing before the pelt starts to decompose. With 4 to 5% Na_2S, correct unhairing is obtainable. A certain degree of alkalinity, which is maintained by $Ca(OH)_2$ in a lime-sulfide liquor, is necessary. Hair removal with Na_2S only is mainly a matter of hair decomposition. The point most sensitive to attack is 1 to 2 mm. above the grain. The hair stumps are not dissolved unless the time is considerably extended and mechanical friction is employed. Best results are obtained at high concentrations, and at 37°, provided that the time is limited strictly to that required for unhairing. Hide substance losses are then less than at lower concentrations with extended time. The swelling caused by Na ion is greater than by Ca ion. Unhairing in weak Na_2S is of interest only if the hair is to be preserved, in which case the temperature should not exceed 20°. In lime-As_2S_3, the alkalinity is even lower than in plain lime. At least 1% As_2S_3 is required to show increased speed of unhairing, compared to plain lime. Rate of unhairing is less than in lime-NaHS, but the system gives a very fine grain, with little swelling. Losses of hide substance range between those for lime-NaHS and lime-Na_2S. The low alkalinity permits use of temperatures up to 30°, especially since this type of unhairing generally is used for small skins that are wanted supple and stretchy. Addition of dimethylamine raises the alkalinity of the bath, and speed of unhairing increases up to 30°. At lower concentrations, dimethylamine has about the same accelerating effect as NaHS or As_2S_3. The gain in time over plain lime is about 2 days. The effect of 1% NaHS or As_2S_3 is much greater than that of 1 or 2% dimethylamine. Hide substance losses in lime-dimethylamine are about the same as in plain lime up to 30°. The advantages of dimethylamine are its mellow action and the fine grain produced. T.D.B.

Automation in the Tanning Industry. J. Mersch. *Halle aux Cuirs* (1956), No. 4, 27—Reading about automation most tanners think, "This is for Ford but not for me. Our raw material is too variable and our lots are too small." This must not be the attitude of an industry in the 20th century. Instead of saying, "This is not applicable", one should think, "Here is something new. How can I use it?" First, materials handling should be studied, then automa-

tion of operations, progressing to automatic controls, and finally to mechanized selection, the favorite domain of electronics. On the road of progress, one would meet naturally the two fundamental conditions of automation: homogeneity of material and large packs. But, seeing more clearly the goal, would it not be of interest, in order to improve homogeneity, to eliminate hides and skins of inferior quality and to process only the top grades or to relinquish parts of the hides which are troublesome or not valuable, possibly by cutting regular shapes to facilitate machine work? It may be necessary to reorganize factories to facilitate processing of larger production units so that automation becomes profitable enough to justify the necessary capital expenditure, and to change recruitment of the working force and reform it in accordance with such new tasks. T.D.B.

Desmophen-Desmodur Lacquers in Leather Finishing. W. Speicher. *Rev. Techn. Inds. Cuir*, **48,** 161 (1956).—The film formation is made by chemical reaction of 2 components of relatively low molecular weight which by themselves are not film-forming but in drying form a polyurethane film of high molecular weight. The reaction is a polymerization of a di- or polyfunctional isocyanate (Desmodur) and a di- or polyvalent alcohol (Desmophen). Types of Desmodur and Desmophen as manufactured by Farbenfabriken Bayer are described as well as their application. Only solvents which do not react with isocyanate groups can be used, like esters, ketones, and aromatic hydro-carbons. For coloring, dyes soluble in organic solvents may be used. It is claimed that the use of Desmodur-Desmophen overcomes the disadvantages of linseed oil and nitrocellulose lacquers. T.D.B.

The Structure of Elastin. V. Moret and L. Gotte. *Boll. Soc. Ital. Biol. Sper.*, **32,** 231 (1956); *Chem. Abstr.*, **50,** 15652*g*.

Infrared Spectrophotometric Observations on Collagen Tissue and Reticular Connective Tissue. M. Scrocco and E. L. Benedetti. *Riv. Istochem. Norm. e Patol.*, **1,** 339–43 (1956); *Chem. Abstr.*, **50,** 15661*b*.

Tannin-like Substances. I.—The Enzymatic Oxidation of Various Phenols. B. Schmidt and H. V. Guttenberg. *Pharmazie*, **9,** 212–20 (1954); *Chem. Abstr.*, **50,** 15749*f*.

Trivalent Chromium Salts. A. V. Pamfilov and N. N. Puchkova. *Zhur. Obshchei Khim.*, **26,** 955–57 (1956); *Chem. Abstr.*, **50,** 15308*d*.

New Methods for the Determination of Sulfur (Sulfide and Sulfhydrate). I. J. Dick and V. Bica. *Acad. Rep. Populara Romine, Basa Certetari Stiint. Timisuara, Studii Certetari Stiint.*, **Ser. I, 2,** 1030–38 (1955); *Chem. Abstr.*, **50,** 15340*e*.

The Aging of Collagen. F. Verzar. *Helv. Physiol. et Pharmacol. Acta*, **14,** 207–21 (1956) (In German; English summary); *Chem. Abstr.*, **50,** 15622*c*.

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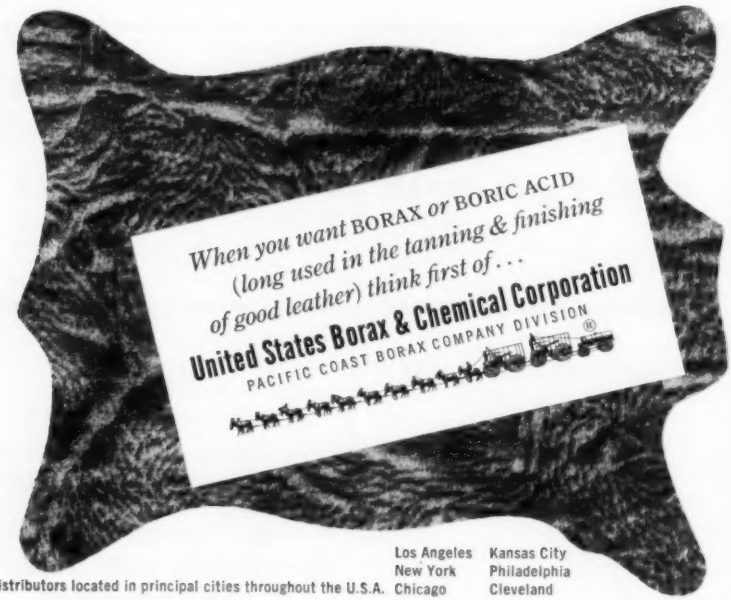
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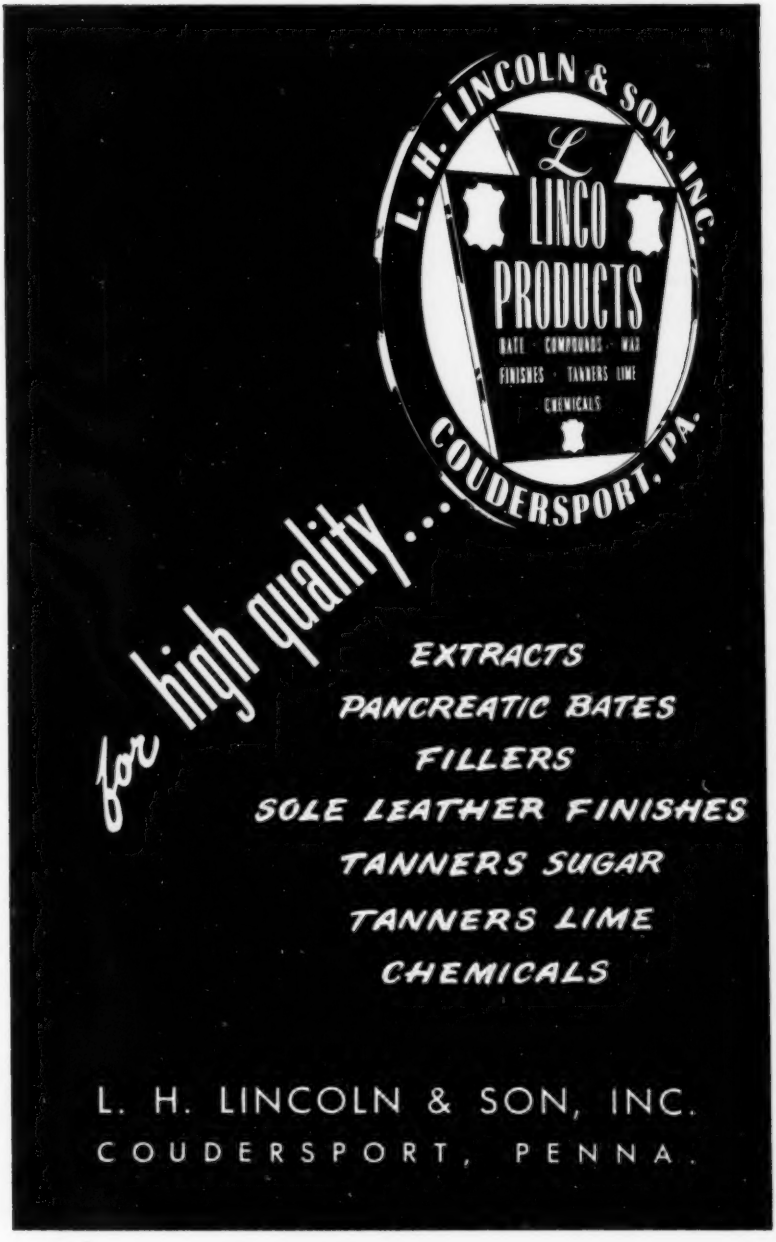
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Non Tannins	16.80
Insolubles	1.10
Water	23.40
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Soluble Solids	75.50

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